



Manufacture of Lunar Regolith Simulants

D.L. Rickman

Marshall Space Flight Center, Huntsville, Alabama

S.A. Wilson and D.B. Stoeser

United States Geological Survey, Denver Federal Center, Denver, Colorado

M.A. Weinstein

Zybek Advanced Products, Boulder, Colorado

J.E. Edmunson

BAE Systems, Inc., Huntsville, Alabama

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National Aeronautics and
Space Administration

Marshall Space Flight Center • Huntsville, Alabama 35812

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LIST OF ACRONYMS AND SYMBOLS

Al	aluminum
Al ₂ O ₃	aluminum oxide
ASTM	American Society for Testing and Materials
BoR	Bureau of Reclamation
βTCP	beta-tricalcium phosphate
Ca	calcium
CaCO ₃	calcite
CaO	calcium oxide
CaSO ₄	bassanite
CIPW	Cross, Iddings, Pirsson, Washington
CMC	carboxymethyl cellulose
Fe	iron
FeOx	iron oxide, oxidation state unspecified
FeS	troilite
FeS _s	pyrite
FPA	fine particle analyzer
GRC	Glenn Research Center (NASA)
H ₂ O	water
H ₂ SO ₄	sulfuric acid
ISO	International Organization for Standardization
K	potassium
KSC	Kennedy Space Center (NASA)
LASP	Laboratory for Atmospheric and Space Physics
Mg	magnesium
Mg ₂ SiO ₆	orthopyroxene
Mn	manganese
Na	sodium

LIST OF ACRONYMS AND SYMBOLS (Continued)

NaCl	sodium chloride
Na ₂ O	sodium oxide
ORBITEC	Orbital Technologies Corporation
OH	hydrous
P	phosphorus
pH	measurement of how acidic or basic a substance is (ranges from zero to 14)
PPI	Plasma Processes, Inc.
PS score	particle size score
RNA	ribonucleic acid
SBIR	Small Business Innovative Research
SEM	scanning electron microscope
Si	silicon
SiO ₂	silicon dioxide/silica
Ti	titanium
TM	Technical Memorandum
USGS	United States Geological Survey
WHIMS	wet, high-intensity separator

TECHNICAL MEMORANDUM

MANUFACTURE OF LUNAR REGOLITH SIMULANTS

1. INTRODUCTION

The lunar regolith, sometimes referred to as lunar soil, is the unconsolidated blanket of material that covers most of the lunar surface. The geology of the Moon can be divided into two main provinces: highlands (leucocratic gabbroic) and mare (basaltic). In addition to the differences between the two provinces, there also are significant variations of the geology within the provinces. The regolith that develops within these provinces reflects both the compositional differences between the provinces and the local variations. Because of lateral transport by impacting, there is also some level of mixing within and between the provinces. The regolith is therefore highly variable.

Any equipment that contacts the lunar surface will interact with the regolith. Because of its abrasive and adhesive properties, the lunar regolith is a serious hazard to astronauts and their equipment. In order to design and engineer the equipment needed for lunar surface landings, excursions, and stations, regolith simulants are required. Regolith simulants can be manufactured for general use or designed to simulate specific properties of the regolith.

There are many ways to make bulk lunar regolith simulants. Some are made by grinding a single rock material, as was done to make the JSC-1 and JSC-1A, and FJS-1 regolith simulants. Other simulants have been made from mixtures of geologic materials and manufactured components, e.g., OB-1 and multiple simulants from Japan. In other cases, especially for specialized applications, waste products from quarries or mines can be used, e.g., BP-1^{1,2} and GSC-1³. And in other situations, mixtures of materials having little similarity to lunar regolith may be created, as was done with GRC-1,⁴ in order to simulate a specific physical property. Normally, the approach chosen will, to a large extent, reflect the uses of the simulant and the user's budget. The specific processes are also a function of total mass being produced in a single production run and the required level of uniformity within and between single runs.

A large number of lunar regolith simulants have been made in the last several decades. For the most part, little to essentially nothing is known about how each simulant was made, either because the manufacturer did not supply documentation or the process was proprietary. The result has been that, each time a new simulant is made, the producer has to invent a process, replicating what was previously learned elsewhere but never reported. This Technical Memorandum (TM) is intended to give at least basic information about methods useful for simulant production. It is based in large part, but not exclusively, on work done by the United States Geological Survey (USGS)⁵ team in support of the simulant development project at Marshall Space Flight Center from 2006 to 2011.

In contrast to the previous production of regolith simulants, the objective of the work reported here was not to create and supply simulants but rather to develop and document the processes involved in creating simulants. Therefore, this TM is much more than a 'recipe'; it also discusses such things as terminology, options, rationales, blind alleys, safety, measurement, and known limitations. The materials to be produced were to be as close to actual regolith as practical without consideration of the simulant's uses.

The primary audience for this TM is someone interested in making a simulant of a regolith found or assumed to be present on a nonterrestrial, solid surface. The authors have assumed the reader is a qualified specialist or a team of professionals seeking sufficient detail to guide the work. A secondary audience for this TM are those who need to understand something of the complexities involved in making a simulant, but are neither geologists nor experienced in mineral processing.

2. FRAMEWORK AND TERMINOLOGY

In order to actually make a simulant, the producer needs to know, be given, or already possess several things. He or she must have a clear understanding of the intended uses of the simulant, a design, identified feedstock source materials, and knowledge of relevant processes to be used in producing the simulant. As a knowledgeable reader will quickly realize from the following brief discussion, design and source material selection are both topics that require highly specialized knowledge. The training required to competently perform these tasks normally falls outside the knowledge base of individuals competent to perform or direct the production processes.

2.1 Simulant Design

Designing a simulant requires consideration of several things. For simplicity here, these will be categorized into three subjects: knowledge regarding use, cost, and availability of source materials. For an example of simulant design document, see Stoeser et al., “Design and Specifications for the Highland Regolith Prototype Simulants NU-LHT-1M and -2M.”⁶

2.1.1 Functionality and Use

To design a simulant that reproduces the engineering features of interest, one must understand the nature of the regolith being emulated and which aspects of that original affect the engineering features of interest. This incorporates several extremely difficult problems as follows:

- (1) What will the simulant be used for?
- (2) What physical and chemical processes are involved in a simulant’s use?
- (3) How sensitive are those processes to differences between the test environment and the actual deployment?
- (4) How are those processes characterized and quantified?
- (5) Where on the Moon is the actual deployment?
- (6) What are the physical and chemical characteristics of the regolith at that location?
- (7) Which of the regolith characteristics affect the processes in (2) above?
- (8) How much variability is there in the regolith with regard to those characteristics?
- (9) How sensitive are those processes to variations in the regolith?
- (10) How closely can the simulant be made to reproduce the characteristics of the regolith?

In practice, the authors have found that with the exception of item (1), there are severe limitations regarding what is usefully known in answering the other questions. This lack of scientific and engineering knowledge makes simulant design heavily dependent on skill and professional judgment. A thorough professional creating a simulant will also know in detail what features of the original are not faithfully reproduced. As the vast majority of simulant users are not qualified geologists, sharing information on the limitations of the simulant is a substantial responsibility of the producer.

2.1.2 Cost

A substantial constraint in simulant design is cost. Simulants are inherently engineered materials. As with any engineered material, the performance of the simulant is normally strongly correlated with cost, which is commonly expressed in dollars per ton. Cost for useful simulants may range from approximately \$10/ton to well over \$50,000/ton. The former can be achieved when using bulk or waste material produced for another purpose and user constraints on performance are relatively lax, or cost sensitivity is extremely high. For example, the BP-1 simulant² falls into this category. Where factors such as inclusion of agglutinates, attention to the calcium content of the plagioclase, avoidance of hydrous minerals, and the composition of the glass is significant, higher cost per ton can be expected.

2.1.3 Source Materials

Three major factors dominate the choice between multiple, potential source materials: (1) The composition of the primary or original minerals in the rock, (2) absence of hydrothermal alteration or nonlunar minerals, and (3) physical access. With respect to composition, depending on the uses of the simulant, finding suitable feedstock material can be relatively easy or very difficult. Where an ordinary terrestrial basalt or basaltic ash is acceptable, there are hundreds of quarry operations in the United States that can provide acceptable material. But as one adds or tightens requirements on the simulant, suitable candidates for simulant production become rare. For example, if a lunar simulant must have the correct melting point, minimal secondary or nonlunar minerals, orthopyroxene-to-clinopyroxene ratios characteristic of the Moon, olivine with normal lunar values of magnesium (Mg)/iron (Fe), and a high glass content, no single terrestrial source can meet these requirements. Therefore, as requirements related to composition become more important, compromise candidates must be considered. With respect to the second point, most terrestrial rocks incorporate any number of minerals that can cause various problems for users of the simulant. Especially problematic are the hydrothermal minerals, those that incorporate either hydroxyl (OH)⁻ or H₂O in their structure. Also problematic are secondary quartz (SiO₂), or very hard minerals, such as garnets, associated with regional metamorphism. Finally, it is one thing to pick up tens of pounds of rock from the backcountry or a hundred kilograms from a road cut. It is a different problem when one is acquiring multiple tons of rock. Physical access, preferably with heavy equipment availability, is essential.

A technical discussion of source selection is far beyond the scope of this TM, and to do the necessary work requires substantial and appropriate training in geology.

Reference is made throughout this TM to materials provided by or obtained with the aid of the Stillwater Mining Company. The company operates the Stillwater Mine near Nye, Montana. The rock complex they mine is part of the Stillwater layered mafic intrusive that has a compositional range that makes a reasonable starting point for emulating the mineralogy of the lunar highlands regolith. The host rock also has relatively little hydrothermal alteration. Critically, the company was willing to provide both access and support. Other source materials used in this work were obtained by various methods, mostly by direct purchase. These will be noted as appropriate.

2.2 Processing—Rock Grinding Concepts

Regoliths formed in a vacuum largely consist of broken rock, which has not been weathered by atmospherically mediated processes. The lunar rocks are igneous and are for the most part in the mafic to ultramafic range, i.e., basaltic and gabbroic. See Stöffler et al. and Rickman et al. for definitions of these terms.^{7,8} To make a simulant of a given broken rock, one can either assemble chemical feedstocks and synthesize them into rocky materials or take natural rock and break it. Synthesis of the relevant minerals is a high-temperature ($>1,500\text{ }^{\circ}\text{C}$) process. It is therefore energetically expensive. For this and other reasons, the latter option—breaking terrestrial rock—is the path most producers of simulants follow. The relevant expertise for the breaking of rock in lots greater than a few hundred kilograms is found in the mining industry. Therefore, some knowledge of the concepts and vocabulary used by that community is necessary to understand simulant production technology.

Comminution is a process in which solid materials are reduced in size, normally by application of external force. Sizing is a process that separates particles by size. Whatever the comminution process, it is commonly coupled with a sizing process such that only particles below a certain size are passed to a subsequent process. Together, the comminution and sizing form a logical unit. The unit illustrated in figure 1 is the simplest unit. Other, more complex units are practical. For example, the sizing process can have a ‘slimes’ output, in addition to the oversized and undersized outputs shown in the figure. Slimes are particles sufficiently small or otherwise special that they interfere with subsequent processes for any of a number of reasons.

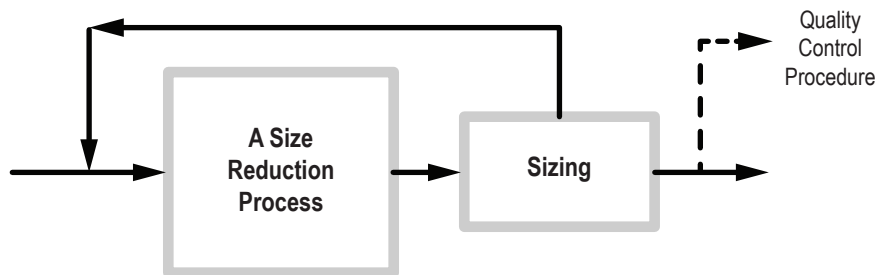


Figure 1. The basic unit of a grinding circuit consists of a method to reduce particle size and a method to assure particles leaving the unit are smaller than a given size. Oversized particles are returned to the grinding process, or in small-quantity, low-value batch processing, the oversized might be disposed of. Quality control, if employed, is commonly neither automatic nor continuous.

A grinding circuit is a combination of one or more of these basic units. The complexity of a grinding circuit is a function of many variables, including the following:

- The nature of the material being processed.
- The size or sizes to be produced.
- The purity of the products to be produced.
- The cost of energy.

There is a vast range of commercially available methods for both size reduction and sizing. In addition to the factors affecting grinding circuit complexity, the choice of technology used in a specific situation is strongly affected by the amount of material to be processed per unit time. Different systems are readily available that can process tens of grams or thousands of tons per hour. Typical secondary factors are the cost of consumables and wear on the components. In some cases, including the production of lunar simulants, introduction of deleterious materials, such as water or cross contamination between grinding runs, can also be factors in the choice of technologies.

In mining, the first size reduction process is typically termed crushing. This is commonly done using a machine called a jaw crusher. Alternative technologies include cone and roll crushers. Subsequent stages in the comminution circuit are often termed either grinding or milling. The most common technologies used for this place the material in a mill containing a grinding media which has a high specific gravity and hardness, such as steel, corundum, or zirconia. Motion of the grinding media on impact with the rock or mineral particles breaks the bonds holding the minerals together, reducing the particle size of the individual grains. The final particle size is determined by factors such as starting sample particle size, mill geometry, grinding time, the sample/grinding media ratio, mill speed, and grinding media density. Alternative technologies can use any of a large number of other approaches. These mills can be operated in either a wet or dry grinding process in both a batch or flowthrough mode. Crushing is normally done dry, and subsequent steps are commonly done wet.

2.2.1 Special Considerations for Simulant Production

For production of lunar simulants, there are several factors that are not normal considerations in the mining industry. Especially significant can be the use of water in milling. It is common practice in milling past the first crushing to add water to make slurry. Where bulk properties of the simulant dominate in the performance of a user's test, this is generally not a concern. For example, total mass, particle size distributions, and load-bearing capacity will not be affected by the use of water in the grinding process. But where oxidation and hydration states of the minerals on the surfaces of the particles is a consideration or the amount of adsorbed H_2O is important, use of H_2O in milling can be a serious consideration.

Another very unusual aspect of simulant production is the need to have a specified particle size distribution. Comminution is almost always controlled by pass-no pass criteria. In most cases, if the particles are fine (small) enough, they pass to subsequent processes. In some cases, if they are too fine, they are also separated. But this logic does not inherently support the production of material with a specific profile of particle sizes.

In practice, this is addressed by making multiple milling runs and varying the milling conditions in each run, especially the time at each step. Alternatively, separate runs can be made with different grinding media or completely different milling techniques. The products of the separate runs are then mixed to produce the desired size distribution profile. As typical milling techniques do not produce uniformly sized particles, the result is a mixture of more or less broad populations. This process makes the skill of the simulant processor very important as the conditions for each step have to be experimentally determined. Further, each time a different source material is used, its specific milling characteristics have to be determined for the milling techniques being used by the producer.

An important consideration when using this type of material processing is that the final product produced will be contaminated to a degree with the grinding media used. For example, the milling process at the Stillwater Mine consumes approximately 1 kg of steel for every metric ton of ore processed (Personal Communication, L. Braunbeck, October 23, 2007). The steel is lost through abrasion between the ore and steel balls used as part of the grinding process. Some of the steel becomes part of the material processed further. Gustafson reported that tungsten, nickel, and cobalt increased in the JSC-1A dust simulant with grinding in a tungsten carbide-lined mill.⁹ While such contamination may be small relative to the total mass of the material, it may be significant in some applications. For example, the magnetic properties of the final material may be affected.

There may be specific physical properties that are adversely affected by prolonged grinding. One specific example is particle shape. The physical processing of geologic material typically results in the 'rounding' of the sharp edges formed when minerals break. This rounding may dramatically affect the cohesion properties of material relative to the starting material or when compared to the same material processed differently. There are marked differences in the flowability of various simulants^{10,11} and figure 2. While there are composition differences between the simulants, flowability is dominated by differences in particle shape and size distributions.



Figure 2. Comparison of the flow characteristics of simulants. From left: JSC-1A <45 µm, JSC-1A >45 µm, JSC-1A, NU-LHT-2M, OB-1, and Coco Beach Sand. NU-LHT-2M did not flow through the orifice under the test conditions. Also note the difference in adhesion as can be seen in the amount of discoloration in the upper chamber. Image provided by Robert P. Mueller of KSC.

Milling can also change a material's chemical properties; this can be caused by increased surface area and surface oxidation of minerals at the moderate temperatures associated with most milling processes. Significant energy is required to break the various chemical bonds holding rocks and minerals together. In the presence of oxygen and water, it is not surprising that chemical changes can and do occur. Some minerals, such as pyrite (FeS_2) are especially susceptible. It will react and form hydrated iron sulfates and sulfuric acid (H_2SO_4). The significance of this is generally minor, but it needs to be borne in mind.

In mining operations, the chemistry of milling products is normally altered while milling. This is done by adding various compounds to the slurry and is done to elicit or suppress specific behaviors needed to separate the valuable constituents from the waste material or avoid selected contaminants. For example, in the Stillwater mill, as part of the flotation process, a total of approximately three-fourths of a kilogram of collectors, depressants, and frothers are added to each ton of rock that goes through the mill (Personal Communication, L. Braunbeck, October 23, 2007). In some mills the slurry pH may be altered. None of the chemicals used are 'lunar.'

Finally, the antecedents of the material used in a simulant need to be considered based on weathering processes. For example, BP-1 is recovered from the waste disposal of a quarry operation in Arizona. In addition to calcite (CaCO_3), it contains bassanite ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$) and halite (NaCl).² All three are nonlunar minerals and could have serious implications to anyone attempting to use the material to chemically simulate the lunar regolith. An ordinary analysis might not have detected the presence of weathering-related minerals.

2.3 Processing—Mixing and Splitting

Once the particles have been ground to the desired size distributions, two major mechanical processes remain—mixing and splitting. Mixing combines the disparate grinding runs, if any, and the various components, if more than one, into a uniform mixture. A wide variety of commercial blending equipment is available. These may include, but are not limited to, cube, cone, V-type, or barrel blenders. The cost of this equipment will vary based on the blending speed, blender volume, and material used in blender manufacture. A consideration in the type of blender used is the potential level of contamination from the blending operation. Abrasion of the blender sidewalls in contact with the simulant material can be significant if prolonged blending periods are used. Splitting produces containers of simulant of uniform composition. Containers ranging from 50-mL glass jars to 1-ton sacks have been used for this purpose. A very common size for many users to request is a 5-gal bucket. A variety of procedures can be used to produce subsamples of a larger supply. Each of these procedures has advantages in terms of cost, equipment needed, time, and sample consistency. When bottle-to-bottle consistency is the most important consideration, the optimal splitting procedure is done using a spinning riffler. In this procedure, the supply of starting material is distributed into a series of containers which rotate in a circular motion beneath the exit port of the sample reservoir. This procedure ensures that multiple aliquots of the starting material are distributed into each container throughout the splitting period. This minimizes sampling bias and facilitates the distribution of mineral phases throughout the sample population.

All parties should explicitly recognize that achieving and maintaining simulant uniformity inherently contradicts the nature of the actual lunar regolith. The regolith is quite heterogeneous within a single sample as a function of particle size, vertically at a single site, and between sites.^{12,13} That uniformity in a simulant is necessary is an artifact of the need to analyze and compare test results obtained using the simulant.

Like grinding, both mixing and splitting are conceptually simple operations, and both are fraught with significant technical hurdles. When mixtures of particles are moved in a gravity field and in the presence of a fluid (liquid or air), the mixtures tend to segregate based on the size, shape, and specific gravity of the particles. The tendency for segregation is very strong when working with simulants (fig. 3).¹⁴



Figure 3. Segregation and stratification in NU-LHT-1M after handling.

To illustrate, NU-LHT-2M has abundant particles $<10\text{ }\mu\text{m}$ and up to 1 mm in size. The mineralogical heterogeneity of the simulant is readily apparent on close examination (fig. 4). The specific gravity of major mineral constituents in NU-LHT series simulants ranges from approximately 2.7 (plagioclase) to 4.3 (olivine). Minor constituents, such as chromite, have even higher values (4.7). Rickman and Street provide a table including specific gravities of major lunar minerals, which can reasonably be expected to occur in simulants.¹⁵ In contrast, the specific gravity of agglutinitic particles can be substantially below that of plagioclase due to their vesicular nature. For an introduction to the problems of handling powders with the intention of obtaining representative samples, see Jillavenkatesa et al.¹⁶



Figure 4. Photomicrograph of NU-LHT-2M in plain light. The large particle in the lower left is approximately 1 mm long.

To avoid or minimize the spontaneous segregation that occurs when mixing and splitting, a number of techniques are used. This is an area that the producer of simulants must address and the purchaser of simulants should explicitly verify if the use is sensitive to variations. If the simulant material is to be shipped, it is best to remix the contents of the bottle using a horizontal or end-over-end tumbling process for several minutes. This mixing procedure should also be used when containers are stored for several days in the laboratory before any subsampling of the materials is performed. Instructions for appropriate procedures are given in appendix A.

2.4 Processing—What Cannot Be Controlled

According to the ISO standard for the evaluation of a lunar simulant, there are four major characteristics to be measured.^{17–19} At this time, two of the characteristics—particle composition and particle size distribution—are well within the control of the simulant producer. The other two—bulk density and particle shape—are not. Density is variable for any given simulant. It may be substantially adjusted by handling.^{20,21} Therefore, as a general rule, the simulant producer does not attempt to explicitly control density. The absence of production control on particle shape distribution reflects a lack of knowledge in two areas. First, the shapes of lunar particles are very poorly characterized. Second, it is known that the choice of milling techniques can affect particle shape, but necessary work has not been done to establish what the relationships are.

Particle shape is affected by three factors that are to a greater or lesser extent within the control of the simulant producer. First, the choice of constituents is a factor. For example, the basaltic ash used to produce JSC-1 and JSC-1A²² or the material used to make FJS-1²³ does not mill the same way as the crystalline rocks from Stillwater. Also, the direct inclusion of glass beads or agglutinitic constituents will also affect particle shape. Second, particle shape is also affected by the length of time the material is milled. Milling techniques and specific mills have practical limits as to

how small a particle can be produced. Additional time in or through the mill tends to do less and less comminution; instead other phenomena, such as particle rounding or simple heating, become significant. Finally, the technique used for milling and the specific settings of the machinery used can affect particle shape. For example, a mill developed by Zybek Advanced Products, Inc. (2845 29th St., Boulder, CO 80301 <www.zybekap.com>), under a NASA Small Business Innovative Research (SBIR) called the Aerodynamic Impact Reactor can produce a very angular product (fig. 5).



Figure 5. Glass used in the Chenobi simulant after milling with the Zybek mill. The material is highly resistant to slump, presumably due to a very high particle angularity.

2.5 Measurements

As noted above, particle size distribution and mineralogical composition of the simulant need to be controlled by the producer; both present technical challenges.

The abundance of individual minerals can be assessed by many methods, including x-ray diffraction or thin section petrography. The measured abundance of individual minerals obtained by these methods is termed modal abundance. However, the cheapest method to assess changes in composition is to obtain what is termed ‘a whole rock chemical analysis.’ A whole rock analysis measures the abundances of the major or rockforming elements: silicon (Si), aluminum (Al), iron (Fe), magnesium (Mg), calcium (Ca), sodium (Na), potassium (K), titanium (Ti), phosphorus (P), and manganese (Mn). By convention, results of such analyses are usually expressed as weight

percent oxides, for example silicon dioxide (SiO_2), calcium oxide (CaO), sodium oxide (Na_2O), and aluminum oxide (Al_2O_3), although the cation may or may not be present in the rock as that oxide. These data can be converted to normative minerals using the CIPW computation.²⁴ This method models the minerals that would have formed under stable, equilibrium conditions from a melt of the stated composition in a closed system. To the extent an igneous rock formed under the assumed conditions, the normative mineralogy will approximate the modal mineralogy. In practice, the results obtained by this approach should be cross-checked or confirmed by modal analysis if absolute mineral abundances are needed. Where relative abundances are needed, i.e., the control of the production process, such cross-checking may not be needed.

Particle size is highly problematic. While the concept of ‘size’ is intuitively obvious to most people, there is no absolute definition of particle size. Instead, there are methods of measuring size, and each method actually measures something different. The user of simulants and the producer of simulants both need to be aware of this. A good introduction to this topic may be found in Jillavenkatesa et al.¹⁶ or Rawle.²⁵ Figure 6 shows that variations between methods of particle size measurements are readily apparent when working with simulants.

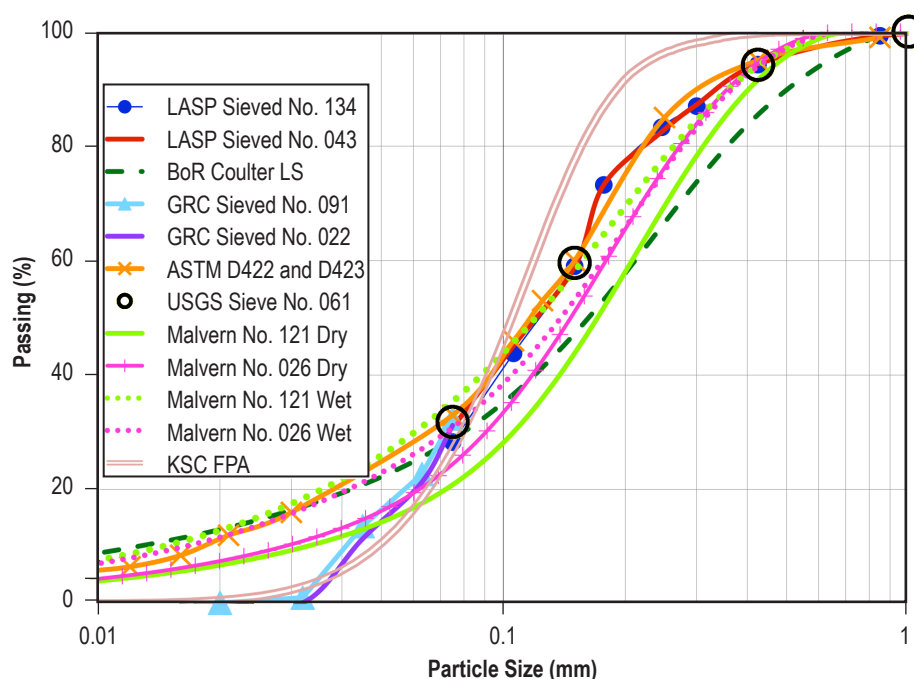


Figure 6. Particle size distributions for NU-LHT-1M determined by different methods. Unpublished data provided as a community effort to characterize the NU-LHT-1M simulant material.

The graph shows particle size distributions for NU-LHT-1M obtained by multiple methods. Each curve is from a different split of the simulant, indicated where known by the ‘No. nnn’ identifier. Four fundamental measurement techniques were used: sieving, hydrometer, microscopic image analysis, and diffraction of light. Not included here are additional size analyses of NU-LHT-1M done by Paul Greenberg using time of flight and motion through an electric field after charging.

Sieving was done by the following: Susan Batiste of the Laboratory for Atmospheric and Space Physics, University of Colorado Boulder, CO; NASA Glenn Research Center (GRC) under the supervision of Kenneth Street; and under the supervision of Xiangwu Zeng at Case Western Reserve University. The data from Batiste and Zeng are for particles $>75\text{ }\mu\text{m}$, and the data from Street are for particles $<75\text{ }\mu\text{m}$. It is noteworthy that the multiple sieve results are highly consistent. This requires that individual samples be highly similar and the methodology to be reproducible between labs.

The hydrometer tests reported by Zeng were done according to ASTM Standard D423,²⁶ which covers particles up to $75\text{ }\mu\text{m}$. A hydrometer utilizes the rate of settling under gravitational acceleration through a liquid.

Image analysis was done by Phil Metzger of NASA Kennedy Space Center (KSC) using a Fine Particle Analyzer. The system passes particles in front of a microscope where image capture hardware records silhouettes of the particles. The raster images are then analyzed for a number of parameters. The parameter used here is the maximum enclosed circle, which is an approximation to the sieve size.

Data from two laser diffractometer systems are shown in figure 6. The data from the Bureau of Reclamation (BoR) were acquired for Steve Wilson using a Coulter LS. The data from Alan Rawle of Malvern Instrument were acquired using a Malvern Mastersizer 200. Laser diffractometers can function based on one or more physical-mathematical models of light scattering. Each model is sensitive to different secondary affects, such as surface roughness, particle shape, and refractive index. The detailed settings and configuration of the Coulter LS used for the measurement reported here is not known. The Mastersizer 200 was set to a refractive index of 1.75, a venturi pressure differential of four bars. It should be noted that NU-LHT-1M is a mixture of phases. For the purpose of a laser diffractometer using Rayleigh, Rayleigh-Gans, or Mie scattering theory, only the refractive indices of the major phases: plagioclase, pyroxene, and glass, are of interest. The approximate refractive indices of plagioclase and pyroxene are 1.58 and 1.67, respectively. The refractive index of the glass used has not been determined but is estimated to be approximately 1.58 based on knowledge of its chemistry.²⁷

As expected, none of the methods give highly similar results over the entire range of sizes. However, over a restricted range in particle size, some methods are very similar, such as the sieving by GRC and the image analysis by KSC for particles $<100\text{ }\mu\text{m}$. A second cluster of measurements over the same size range is formed by the wet laser diffractometer results by Malvern, the Coulter LS, and the hydrometer; the two clusters are quite distinct from each other.

None of the different particle size measurements shown in figure 6 are ‘wrong.’ Each method has its advantages and disadvantages. Because cross comparisons of results between methods are extremely problematic, it is advisable for the simulant producer and the simulant user to agree on a modality of particle size measurement. It is also important that data published on particle size explicitly state the details of how the data were acquired.

3. SAFETY

In the production of lunar regolith simulants, there are many places where the producer needs to act within industry norms for the safe handling of large quantities of rock or mineral powders. This TM assumes the producer is knowledgeable of and follows guidance on such points as appropriate eye and hearing protection, appropriate footwear, lifting protocols, etc. The topic where the producer and the user may need guidance is in safe handling of simulants as respirable mineral dusts. At this time, the topics of potential concern are ‘silica,’ iron sulfides, fibrous silicates, and acute dosage.

3.1 Silica

Silica, which chemically has the formula SiO_2 , is a known health hazard when inhaled as a dust, i.e., particle sizes $<10\text{ }\mu\text{m}$. Chronic exposure to such dust at sufficiently high levels is responsible for an incurable condition called silicosis, which can be fatal. An e-mail from Dr. Kenneth Street, GRC, on November 7, 2007, 1:08:08 p.m., captures the conceptual framework from which most people approach this information:

Subject: RE: Definitive statement: quartz $<0.5\%$ max in JSC-1AF sample

... Many months ago I became involved with the dust project and JSC-1a. I immediately looked at the MSDS and recognized that we had a problem. The MSDS I was examining listed the composition of JSC-1a as containing 46-40% SiO_2 . I also scanned to the bottom of the component list to find Na_2O and K_2O which do not exist on earth because they hydrolyze immediately in any humidity to their respective hydroxides. Even though Na_2O and K_2O do not exist, SiO_2 does and hence the problem of eyes looking at this information and not knowing what it really is. ...

As Dr. Street properly notes, this looks like “an industrial hygiene disaster waiting to happen.” He also points out in his e-mail that it is important to know what the terms mean.

Unfortunately, the term ‘silica’ has many meanings and usages specific to different disciplines. As stated in section 2.5, by convention, whole rock chemical analyses of the major elements are expressed as weight percent oxides. Thus, even though an analysis may indicate, for example, 50% SiO_2 , this is typically combined with the other major elements to form minerals such as feldspar, Fe-Mg silicates, and so on. Thus, such a rock may have no free silica at all and there is no implication from such an analysis that a specific SiO_2 or silica mineral such as quartz exists in the rock. Typically, it takes an igneous rock with greater than about 65% SiO_2 in its analysis before a SiO_2 mineral is actually present. What is needed for evaluation and management of risk to health is knowledge of the actual minerals present and their abundances.

As Dr. Street wrote in the same message, the appropriate analyses must be done according to standard methods by people who are demonstrated to be competent in the analysis. For the analysis of silica, this means use of a ‘Certified Silica Analysis Laboratory.’ Such a lab looks for three minerals: quartz, cristobalite, and tridymite, all of which have the chemical formula SiO_2 . The cost of such analyses in 2012 is <\$1,000. Other forms of silica may also be of interest, such as amorphous silica, and may need to be considered for analysis. The analysis of JSC-1A by the certified lab, RJ Lee Group, Inc., found the following values in weight percent:

- Quartz: <0.2
- Cristobalite: <0.2
- Tridymite: less than detection limit of 0.1.

Feedstocks which have not been explicitly checked for quartz, cristobalite, tridymite, and amorphous SiO_2 should be evaluated by competent and qualified personnel. Quartz is such a common mineral phase, as either a primary or secondary mineral, that assuming its absence is in many cases not prudent. For example, basalts can have a small amount of free silica as either a late stage differentiate or it is introduced later by hydrothermal activity or groundwater. It should also be noted that freshly crushed quartz is more hazardous than material that is ‘aged’ 2 mo.²⁸

BP-1 was analyzed for health risks, especially silica, in part due to the results given in Stoeser et al.² KSC’s Industrial Hygiene Office provided specific handling recommendations based on the material’s use in a Lunabotics contest of excavators created by college students. See Greg Galloway’s report in appendix B.

3.2 Other Minerals

Iron is a very common element in rocks and is a necessary constituent in a very large number of minerals. In the igneous rocks frequently used to make lunar simulants, there can be one or more iron-containing sulfides. These sulfides, such as pyrite and pyrrhotite, have received attention as possibly significant risks to pulmonary health. In addition, iron-sulfide may be added to simulants; e.g., NU-LHT-2M, to simulate the presence of troilite (FeS) that occurs naturally in the regolith. Schoonen et al. discuss some of the relevant research.²⁹ For example, pyrite is known to generate reactive oxygen species in vitro. Pyrite has also been shown to degrade RNA (ribonucleic acid) orders of magnitude faster than quartz. Based on what is known about the processes involved, it seems likely the other iron-containing sulfides may have similar effects. Acceptable exposures to iron sulfide containing respirable dust have not been set as far as the authors are aware. Based on the available information, managing exposure to it, at least to the same degree one uses for quartz and other silica polymorphs, seems appropriate.

Much of the lunar regolith has compositions that geologists considered mafic to ultramafic. Thus, for simulant manufacture, rocks of similar composition may be sought. In certain situations, hydrothermal or metamorphic alteration of the primary, or original, minerals in such rocks can create secondary minerals with fibrous structures. Some of these fibrous minerals are associated with asbestosis, other nonmalignant lung and pleural disorders, lung cancer, mesothelioma, and other cancers. Therefore, when geologically appropriate, sources being evaluated for use in the production of simulants should be checked for the presence of asbestiform minerals.

3.3 Acute Exposure

Simulant producers and users are cautioned that most lunar regolith simulants of the type discussed in this TM will be abrasive to skin, eyes, and an irritant to mucosal membranes. These materials will also contain significant amounts of respirable dust (<10 µm particles). The available information (see Conclusion section, app. B) indicates rare or occasional handling of open containers of simulant poses no appreciable risk, but respirable dust should be avoided or controlled during manufacture, handling, and substantial periods of use.

3.4 Other Health Information

A small amount of work has also been done on the solubility of NU-LHT-1M and NU-LHT-2M in simulated biological fluids.³⁰ The fluids used for their work were simulated lung fluid, simulated phagolysosomal fluid, serum-based fluid, simulated gastric fluid, and simulated sequential gastrointestinal fluid. Some the elements showed very high solubilities. Without more detailed study, the significance of their results is not known.

For an introductory discussion of toxicology of mineral dusts, Plumlee et al. is suggested.³¹ In the same volume, the chapter by Schoonen et al. on “Mineral-Induced Formation of Reactive Oxygen Species” is useful and includes information about silicosis.²⁹

4. MINERAL SEPARATION

The economic pressure to process low quality ores has driven the development of many mineral separation technologies which can separate one or more minerals from the other minerals in the rock. Individual technologies are based on a wide range of physical and chemical properties. In each technology, a desired, economically valuable phase differs from the ‘gangue,’ or waste phases in the ore. Some of the properties used include specific gravity, chemical affinity to specific polar molecules, induced electrical charging, selective solubility, magnetism, and particle shape. Each ore body has its own specific optimum separation protocol. The determination of that protocol is the domain of extractive metallurgical engineering.

For many simulant applications, there are no terrestrial rocks that adequately match lunar regolith. Therefore, using a terrestrial rock as a component in a lunar regolith simulant is necessarily an exercise in compromise. However, all of the major lunar minerals, as well as most of the minor and trace minerals, are readily found on Earth as constituents of rocks. If the desired minerals could be readily separated from their source rocks, the need to closely match rock composition to lunar prototypes would be reduced or eliminated.

Mineral separation has other advantages for simulant production. The processes of separation inherently remove undesired or deleterious phases present in the source rock. The separation processes also tend to reduce variation in the feedstocks for manufacturing. Separation also would permit the usage of feedstocks that have a highly desired, single phase, but whose other phases would be very objectionable. And using relatively pure, end-member minerals produced by mineral separation could make production of a simulant of arbitrary composition a relatively simple matter, even if the target simulant composition was far outside the composition of available terrestrial rocks.

The major downside to using mineral separates is cost. Mineral separation requires grinding the rock until the desirable phase(s) is mechanically liberated from the other phases. The reason for the enormous comminution facilities at mines is to prepare the rock for mineral separation. Reducing the particle size of a rock is an energy-intensive operation and requires equipment designed for that purpose. Following grinding, the separation technology also requires specialized equipment for that specific purpose. Operations of both the milling and the separation processes require trained and skilled personnel. Finally, the cost per ton for mineral separation is very sensitive to the tonnage of material to be processed. The handling of small quantities—hundreds of kilograms to a few ton of rock to be processed—is much different than processing thousands of tons of rock a day.

Thus, a choice whether to use mineral separation is a complex problem. Simulant performance, tonnage required, access to feedstocks, multiple skilled labor requirements, transportation, storage, capital costs, and how individual costs are accounted are all factors in the trade space.

To illustrate, the development of each of the initial NU-LHT series simulants required a trip to the mine by several Ph.D.'s to hand select and load individual stones. The mass collected each trip was several ton. This approach was deemed necessary to reduce the amount of alteration minerals present in the simulant feedstock. A quick, minimum estimate of this single expense (\$200 salary, benefits, and overhead/hr \times 8 hr/day + \$60 per diem \times 3 days + \$1,000 transportation \times three research scientists, is \$17,940 per trip). This expense is only one of several labor elements in the task of feedstock collection. As the justification for using the high-skilled labor, reducing the abundance of alteration minerals, can be achieved by mineral separation, the cost of separation may be competitive if sufficient tonnage of high-fidelity simulant is required.

Multiple experiments were done in mineral separation; two are reported in detail elsewhere.⁸ Section 6 of that report covers dry magnetic separation tests done by Hazen Research and Eriez Manufacturing using 'road norite' from the Stillwater mine as feedstock. Both experiments demonstrated process flows producing high-quality plagioclase concentrates. The quality of the Eriez product was established by x-ray at the USGS. It was measured as being $\approx 99.4\%$ pure. Producing a pyroxene concentrate from this or similar feedstock is also desirable. An incidental result of both the Hazen and Eriez work was a series of partial pyroxene concentrates. Unfortunately, the x-ray analysis of the Eriez pyroxene concentrates were found to contain between 5% and 15% talc+chlorite+hornblende and $\approx 22\%$ plagioclase. Visual examination of the Hazen splits shows it is also impure.

Courtney Young, Department Chair of Metallurgical & Materials Engineering, with colleagues and students at Montana Tech, performed a series of exploratory tests, which included olivine, pyroxene, and plagioclase concentration and talc suppression.³² Among their findings is the observation that electrostatic separation was not successful. They also found that the road norite, when crushed to -30 U.S. mesh ($-600\ \mu$) could be dry magnetically separated to produce a concentrate that was $\approx 98\%$ plagioclase. The $\approx 1\%$ pyroxene contamination in this product was due to particle locking. Thus, their results on this point replicate the earlier results from Hazen and Eriez.

Some of the work done at Montana Tech used the mill sand from the Stillwater Mining Company's mill. For a discussion of the mill sand, see section 6.1. As a feedstock for mineral separation, the mill sand has many significant advantages: it is highly uniform, already reduced to appropriate particle sizes, it is readily obtained, and extremely abundant. The mill sand also presents some special problems: based on the available information, it contains more alteration minerals than the 'road norite,' and it contains various compounds used in the flotation processes of the mill. In one element of their experiments, the Montana Tech team combined wet magnetic separation, chemical cleaning, and selective flotation of the mill sand. This successfully established that the mill sand could be used to produce a concentrate of plagioclase, remove the alteration minerals grouped under the heading of 'talc,' and produce a pyroxene product. Their flowchart is given in figure 7 (redrawn with permission of the author).³²

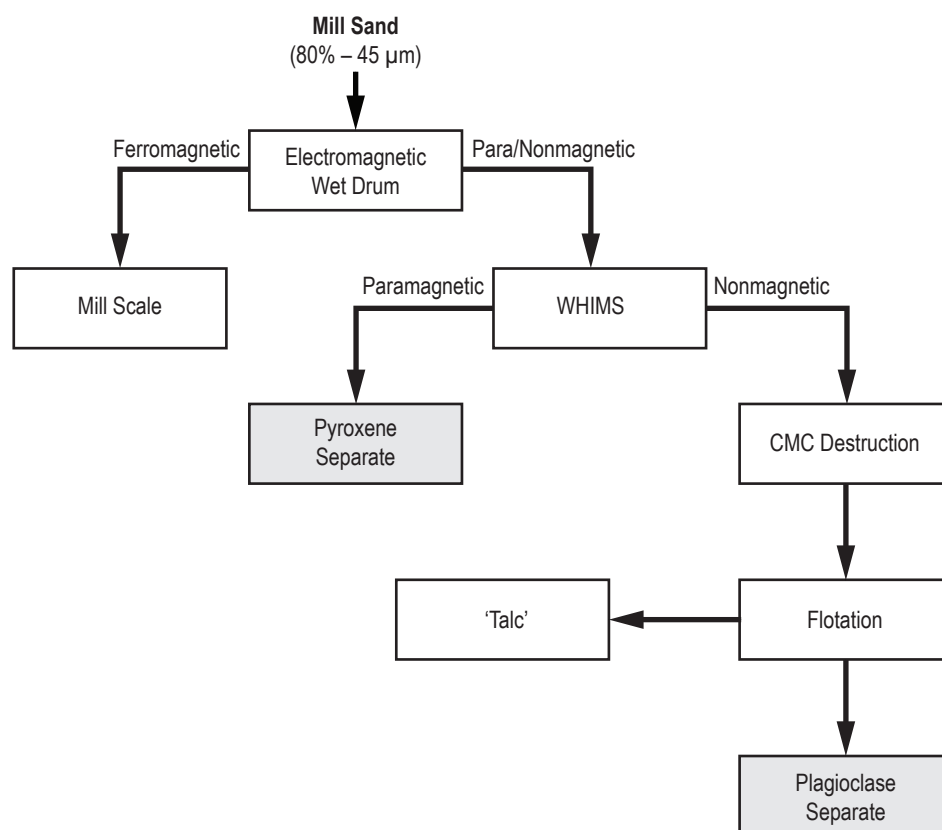


Figure 7. Mineral separation flow chart utilizing magnetic separation and flotation developed at Montana Tech. The feed is Stillwater ‘mill sand’.

The mill sand contains steel, dominantly from abrasion between the ore and the crushing and grinding equipment, which is removed by the electromagnetic wet drum. The less magnetic and nonmagnetic fractions go into the wet, high-intensity separator (WHIMS). The nonmagnetic fraction produced by the WHIMS is dominantly plagioclase, but it also contains various alteration minerals. As some of the alteration minerals can interfere with the recovery of the platinum-bearing phases, the Stillwater mill adds an agent, carboxymethyl cellulose (CMC), to force the talc phases to report to the mill sand. The CMC, which is also used as a food additive, can be destroyed by the addition of hydrogen peroxide. After removal of the CMC, the talc-type minerals can be separated from the plagioclase by flotation using a fatty acid collector. The result is a high purity plagioclase product.

The paramagnetic split produced by the WHIMS in figure 7 has not been fully characterized, but is dominantly a mixture of clinopyroxene and orthopyroxene; it also contains alteration minerals such as hornblende, chlorite, and probably talc. One of the ongoing research problems is how to separate the pyroxenes from each other and from the alteration minerals. Another task is to determine the abundance of and location of observed alteration minerals, including albite, zoisite, stilbite, quartz, FeOx species, amphiboles (hornblende), and calcite.

5. SYNTHESIS

Several phases found in lunar regolith are not practically available from terrestrial sources, or when available, may contain additional, undesirable phases or may not be cost effective. Therefore, attempts to synthesize components was explored.

5.1 Glass

Glass is a major component of the lunar regolith.¹³ It exists as both glass particles and in various admixtures with mineral or rock particles. Simulation of the different regolith glasses is highly dependent on the use of the simulant and of which glass fraction is being considered. For many applications where only the mechanical performance of the simulant is considered, almost any commonly available silicate glass can be used as a simulant for the pure glass particles. If other considerations, such as melting temperature, melt chemistry, melt viscosity, or spectroscopy are significant, more attention to the composition of the glass is essential. Natural glass, obsidian, can be used but is substantially higher in SiO₂ than lunar glasses.

Slag from a copper-nickel smelting operations was used in the OB-1 simulant.^{20,33} This glass has a normative composition rich in olivine, which is typical of slag glasses from such smelters, including that of Stillwater (W. Fitcher, “MLA Evaluation of Stillwater Slags, The Centre for Advanced Mineral and Metallurgical Processing, JKTech Job No. 5295, Montana Tech, Butte, MT, 2005, unpublished). The major element normative composition of the Stillwater slag is roughly one-third each anorthite, pyroxene, and olivine. For some applications, the use of such slags in simulants should be evaluated for the amount and nature of trace phases. These will frequently include such things as metallic iron, copper, and a range of sulfides.

Where the use of the simulant is particularly sensitive to glass composition, there are no suitable and commercially available glasses, either natural or manufactured for other uses. Therefore, custom glass production was developed. This work was done by Zybek Advanced Products, Inc., (2845 29th St., Boulder, CO 80301, attn: Michael Weinstein <www.zybekap.com>) in close cooperation with Steve Wilson of the USGS.³⁴ Zybek had previously developed a patented electric-arc plasma melter capable of rapidly melting particulate mixtures of custom composition to produce glasses in commercially significant quantities.

In the development process for simulant, dried mill sand (see sec. 6.1) is fed into the reaction chamber where the electric current is coupled through the melt (fig. 8). When the molten pool has reached appropriate size, residence time, and viscosity, it is permitted to exit the reactor. The liquid silicate is then allowed to freefall into a water bath where it becomes glass (fig. 9). In the subsequent simulant production of the NU-LHT series simulants, this product is referred to as ‘high-quality glass,’ as it lacks visible inclusions of minerals. For the work reported in section 6, the chemical composition of the glass produced this way closely matches the total composition of the other components in the simulant. For simulant production, this was considered extremely desirable.

The technique developed for the NU-LHT series simulants has also been used in the production of the Chenobi simulant.

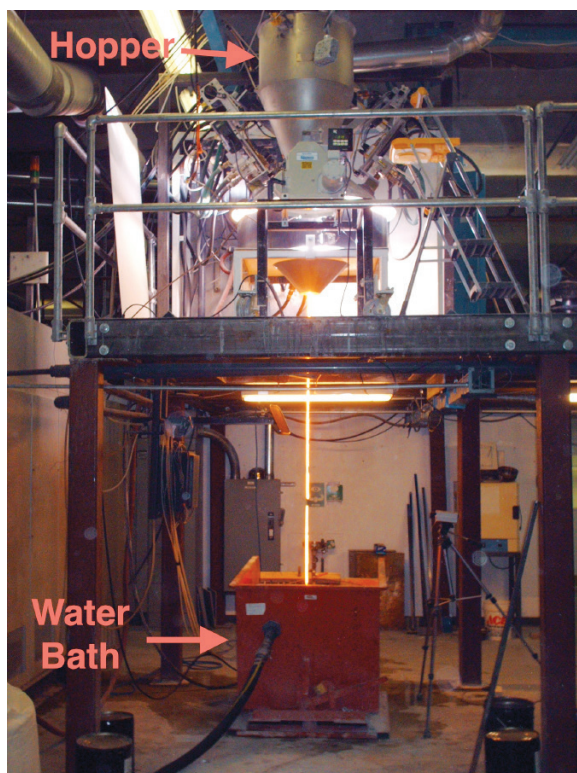


Figure 8. Glass in production at the Zybek Advanced Products facility. Mill sand is fed into the reactor from the hopper. Molten material exits the reactor by gravity and is chilled in the water bath. Note the stepladder in the foreground of the upper level for scale. The height of the water bath container is ≈ 1 m.



Figure 9. Glass produced from melting of mill sand. This product is referred to as 'high quality glass.'

5.2 Agglutinates

Another important glass-bearing constituent of the lunar regolith are particles termed ‘agglutinates.’ These are agglomerates of microrocks, minerals, and preexisting glasses welded together by highly vesicular glass (fig. 10) as a result of micrometeor impacts. They are very delicate cindery particles typically less than a millimeter but can be up to several millimeters long. Agglutinates also contain nanoscopic particles of metallic iron, which is considered a diagnostic feature. This $n\text{Fe}^0$ may affect a range of physical properties.

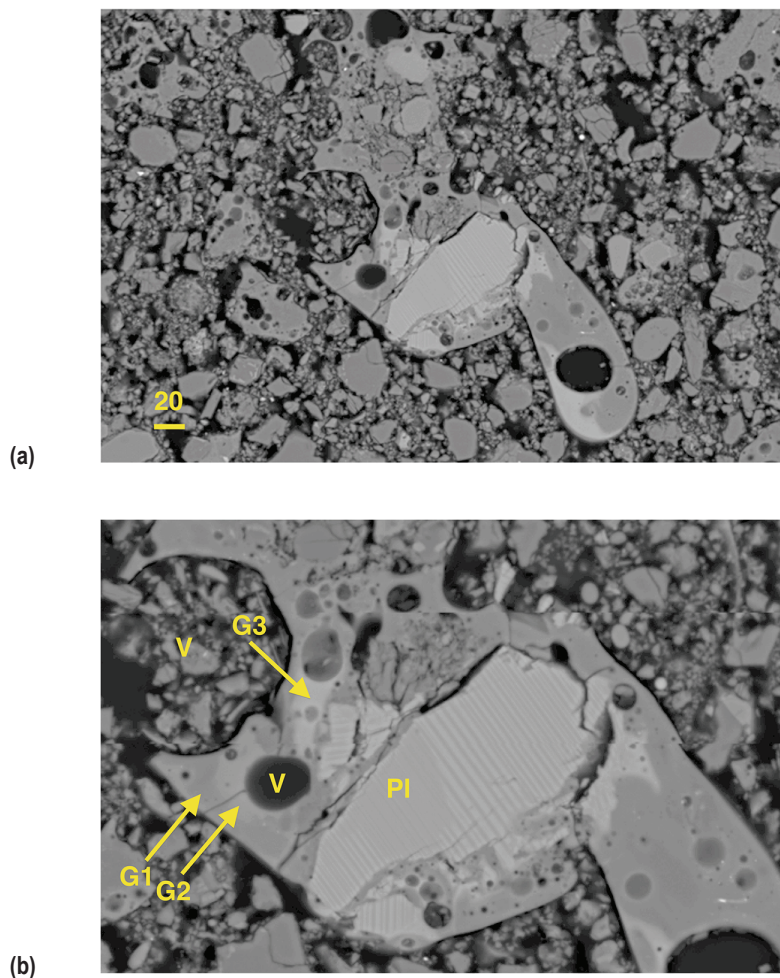


Figure 10. Backscatter electron image of an agglutinate particle from Apollo 16 sample 64001,6031:³⁴ (a) The agglutinate is seen in context of the other particles in the regolith (scale bar is 20 μm), and (b) shows some of the detail in the particle. Example vesicles are marked by V. A particle of plagioclase; exhibiting polysynthetic twinning is marked by Pl. Multiple, preexisting glass phases are marked by G1, G2, and G3. The G3 phase apparently includes a preexisting vesicle above the head of the G3 arrow.

* A.R. Butcher, P.W.S.K. Botha, A. Benedictus, et al., “NASA-USGS-Intellection Apollo 16 Drive Tube Core Characterization Project, Brisbane, Australia, 127 pp., February 1, 2008, unpublished

Replication of agglutinates has been attempted for more than 20 years and is technically challenging.^{35,36} Conditions hot enough to produce a silicate glass tend to rapidly melt and destroy the microscopic particles to be included in the glass. Further, the production of metallic iron as $n\text{Fe}^0$ requires extremely reducing conditions which is hard to achieve in a system that must introduce feedstocks into a reaction chamber while also removing product. Numerous methods have been used to replicate all or some subset of features found in agglutinates.^{37–41}

Two companies have now demonstrated the ability to make high-quality synthetic agglutinates: Orbital Technologies Corporation (ORBITEC) (1212 Fourier Drive, Madison, WI 53717, attn: Robert Gustafson, <www.orbitec.com>) and Plasma Processes, Inc. (PPI) (4914 Moores Mill Road, Huntsville AL 35812, attn: Daniel Butts, <www.plasmapros.com>). Their products contain $n\text{Fe}^0$ in vesiculated, silicate glass or appropriate composition and incorporate mineral inclusions.^{42–45} The materials made by the two companies are the only synthetic agglutinates known by the authors to meet all of the criteria for lunar agglutinates. The two companies' production methods are fundamentally different and patents are applied for. Both product development efforts were in part funded by NASA's SBIR contracts and the methods used are proprietary.

The amount of agglutinate particles in the lunar regolith varies substantially, but may exceed 60% in old surfaces.¹³ The scales of production that ORBITEC and PPI can achieve are in the kilograms per day range. At this time, for most applications, the cost per kilogram of their products is prohibitive for utilization where a metric ton or more of simulant is needed. Zybek Advanced Material Products, continuing the collaboration with the USGS, has developed technology to make a product referred to as pseudo-agglutinates,³⁴ for which a patent has been applied for. This material is a highly vesiculated glass with abundant mineral particles included but does not contain $n\text{Fe}^0$.

The Zybek agglutinate production process utilized Stillwater mill sand material (<1 mm diameter). The sand material was transferred to the USGS where it was dried at 43 °C (110 °F) in a forced-air oven for 24 hr. The dried material was then transferred to a specially modified Zybek twin-screw powder delivery system. The powder delivery system deposited a thin layer of powder approximately 2 mm deep by 10 cm wide onto a rotating steel plate. The plate (90 cm diameter) rotating at approximately 3 rpm moved the powder in a high-temperature plasma zone operating at 2,100 °K (3,300 °F). The plasma was created using a custom-designed, remotely coupled DC plasma. It is estimated that residence time of the powder in the plasma zone was 1–2 s. This brief but intense heating interval created a mixture of partially melted material fused together in a random collection of glass and crystalline components (fig. 11). Synthetic agglutinate material produced in this manner was found to be especially fragile and subject to rapid particle size reduction when subjected to standard grinding procedures.

It was also noted that the production of pseudo-agglutinates was unusually destructive of the manufacturing equipment due to abrasion (Michael Weinstein, Personal Communication, July 13, 2008). Sealed-bearing and lead-screw actuators failed after 6 hr of production at a rate of approximately 50 kg per hour. These components normally last an order of magnitude longer, and produced many more products. It is not known why the difference in wear occurs.



Figure 11. Five-gallon bucket of pseudo-agglutinate produced by Zybek. Visually the material is a mixture of mill sand and vesicular glass in varying proportions.

The complex nature of the pseudo-agglutinates produced by Zybek can be seen in figures 12–14.

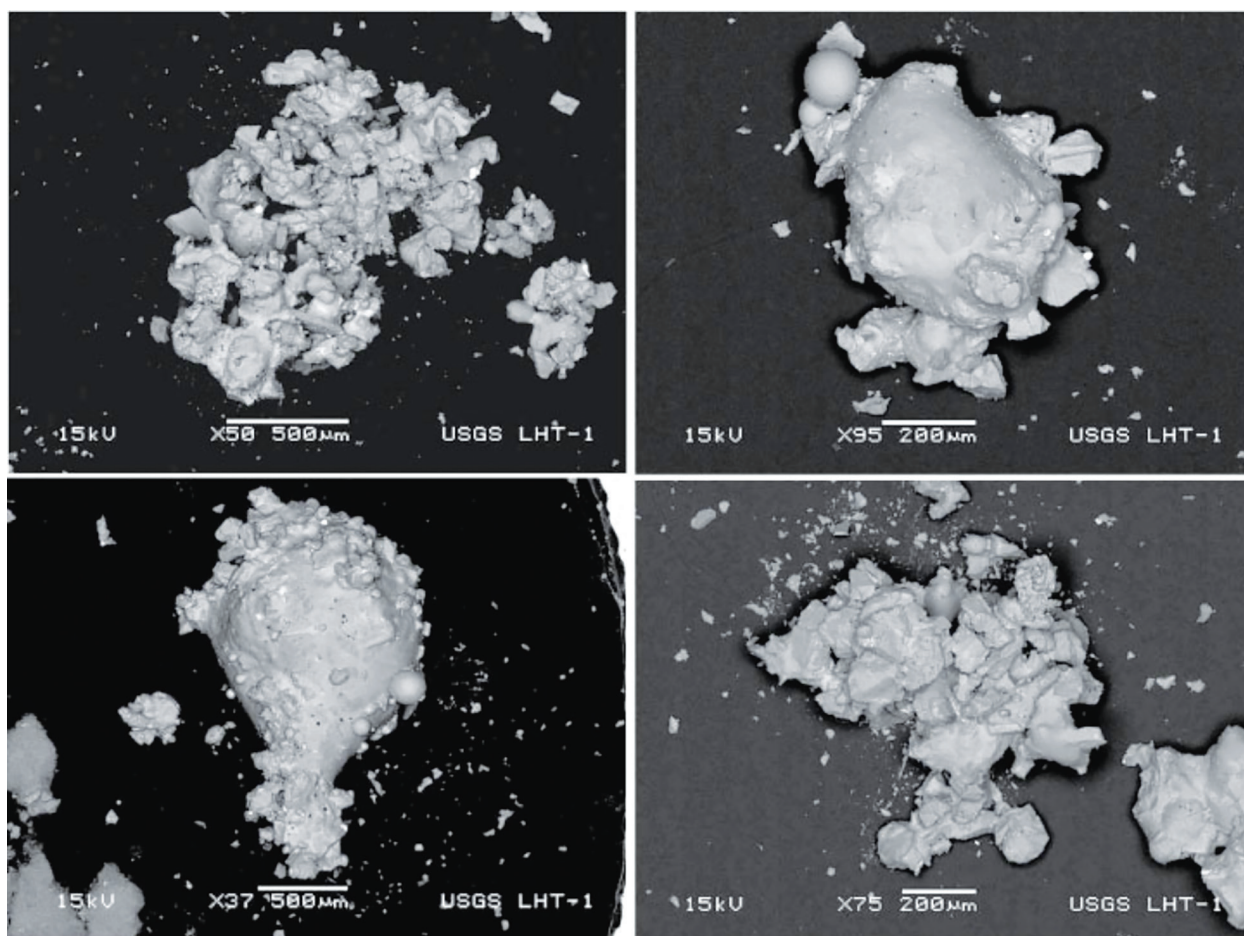


Figure 12. SEM imagery of pseudo-agglutinate particles from the NU-LHT-1M pilot. The individual mineral particles bonded by glass are readily apparent.

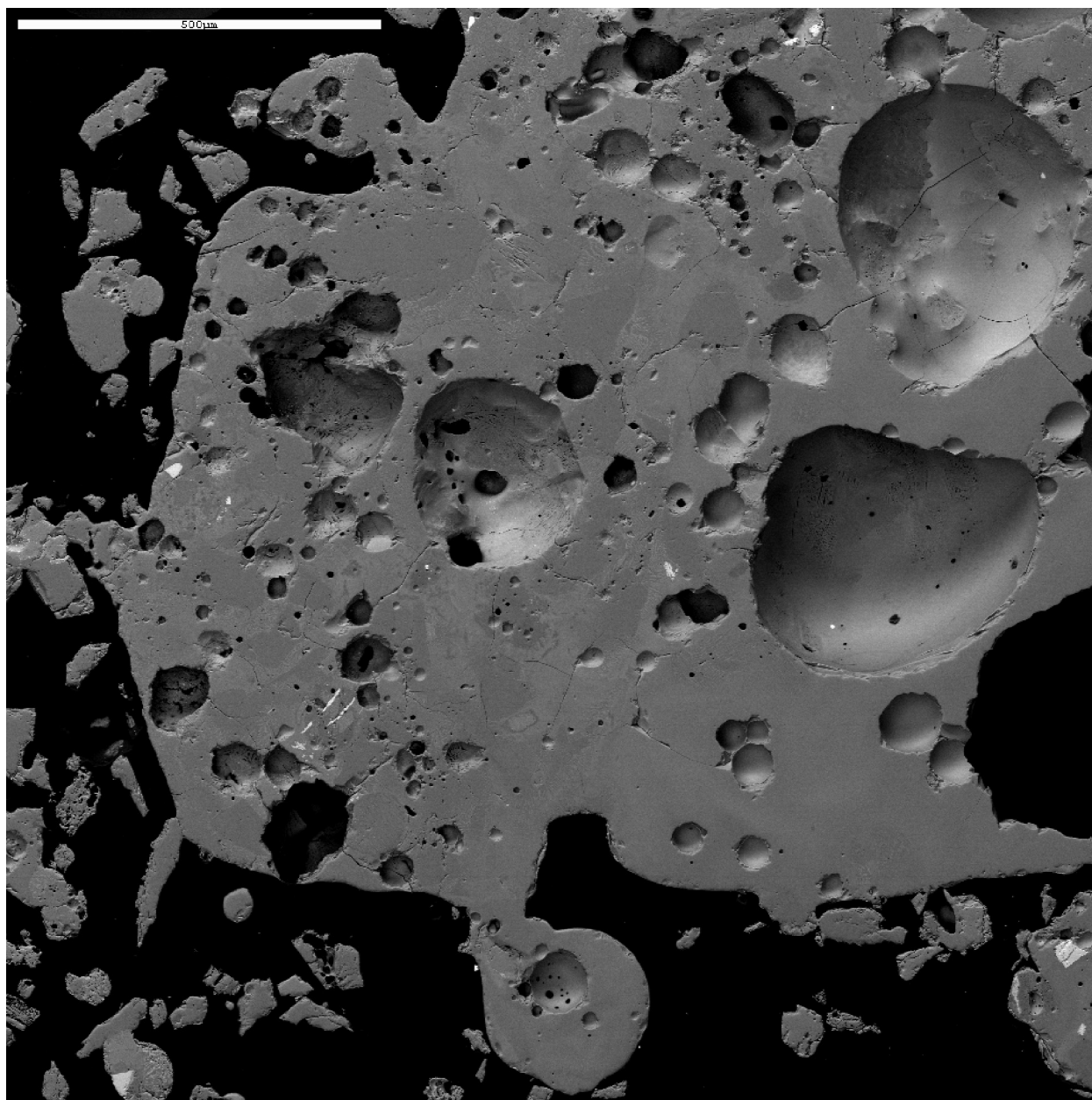


Figure 13. SEM image of a polished section from a pseudo-agglutinate particle. The highly vesicular nature of the material is apparent, as is the variation in internal composition.

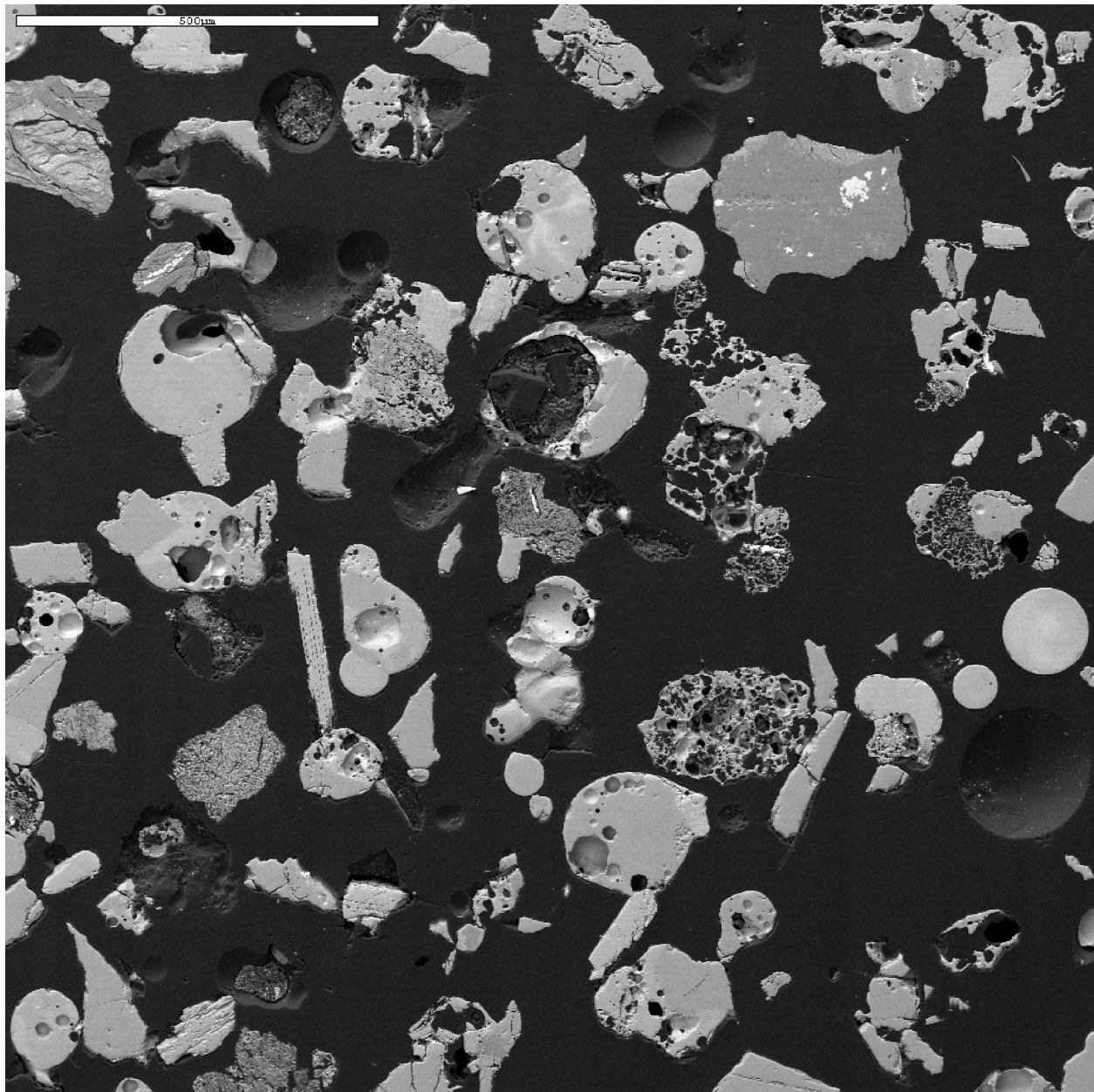


Figure 14. SEM image of a polished section of pseudo-agglutinate and other particles coarser than 60 mesh (250 μm).

5.3 Breccias

A short research effort was undertaken to develop synthetic impact melt breccias using the Zybek plasma technology.³⁴ In general terms, a breccia is a rock comprised of angular fragments (clasts) cemented together by a matrix material. At the Apollo 16 site, over 95% of the rocks on the surface are impact breccias. Impact breccias consist of clasts of earlier breccias, secondary melt derived rocks, shocked primary anorthosites, and feldspathic gabbros in a glassy matrix. The heat necessary to form the glass of the matrix is from a meteor impact. Most of these breccias will be

significantly more friable than a normal crystalline igneous rock, which has important implications in terms of mechanical processing.

Four major components were used in the synthetic breccia. The first component was crushed norite and anorthosite from the Stillwater mine site; this provided plutonic rock and monomineralic fragments. The second component was crystallized (devitrified) noritic material from a melt made from Stillwater sand. This crystallized material is intended to be a partially glassy, lithic analogue of the 'clasts' found in highlands impact breccias. The third component was glass prepared from the Stillwater sand. Each of these three major components was subsequently added to the molten pool of Stillwater sand, the fourth component, creating a mixture of clastic particles in a glass matrix.

The devitrified melt material was prepared by melting Stillwater sand using a static reaction vessel and plasma arc technology. After ≈ 1 hr of melting, the plasma was turned off and the molten 'boule' was allowed to cool at room temperature. It would normally require 2–3 hr of cooling before the boule was removed from the melter. Examining a cross section of the boule revealed that the majority of the material had crystallized into a dark grey material with a wide range in grain size. In addition, there were commonly glass stringers imbedded into specific regions of the boule. It was thought that these glass stringers were aligned with the location of the plasma torch above the molten pool. When practical, the glass stringers and crystallized material were separated and stored separately. Both the glass and devitrified material were crushed to 1–2 cm particles.

The large rotating reaction chamber (skull) was filled first with crushed anorthosite to act as a thermal barrier between the bottom of the skull and the powdered material. Stillwater sand was then added to a depth of 22 to 30 cm. A 5-cm layer of crushed boule glass was then added, and the surface material sculpted by hand to form a slight concave depression in the middle of the skull. The plasma torches were then positioned over the powder and ignited. Within 10 min a molten pool was produced which measured approximately 30 cm in diameter. The heating process was continued for approximately 1 hr during which time the powder level in the center of the skull dropped approximately 10 cm. After 1 hr the plasma was shut off and aliquots of the crystalline norite/anorthosite were added along with crushed devitrified boule material. Even though aggressively stirred with a shovel, the initial addition of clastic material did not sink into the melt as anticipated because the melt was too viscous. A steel rod was therefore used to mechanically mix the molten pool with the clastic material. After a 1-hr cooldown period, the breccia material was removed. Approximately 10 kg of breccia material was made in this initial experiment (fig. 15).



Figure 15. Synthetic breccia. Greenish black glass, devitrified glass (immediately above the penny), and lithic clasts may be seen.

5.4 Minerals

Multiple attempts were made to synthesize two mineral groups for use in lunar regolith simulants. One group was the high calcium end of the plagioclase family. The other group was members of the pyroxene family in the range of lunar compositions. Plagioclase was of interest because the high calcium members are relatively rare in terrestrial, crustal sources, and when found, the hosting rock is not suitable for use in lunar simulants. Synthetic pyroxenes were of interest because there is no commercial source for pyroxenes of appropriate composition.

5.4.1 Pyroxene

The pyroxene family has a complicated phase diagram. For lunar work consideration can be restricted to a ternary system, Ca, Fe, Mg – silicate, which is still a relatively complex system. The first synthesis attempt targeted the mineral augite and was done at the Missouri University of Science and Technology as part of a spring, 2009 Ceramic Engineering 262 course. The students were

Erin DeVries, Nikita Robinson, Anna Trumble, Nathan Rouse, and Casey Slaughter, supervised by Mark Schlesinger. They made two attempts in which they melted and then quenched different melts of different compositions: Trial 1, 20.6 g CaO, 5.1 g Fe, 21.3 g Fe₃O₄, 14.8 g MgO, and 44.1 g SiO₂ and trial 2, 28.6 g CaO, 36.6 g FeO, 20.53 g MgO, and 30.6 g SiO₂. The quenched samples were black and heterogeneous. The quenched samples were subsequently then annealed in air at 850 °C for 72 hr. Neither attempt produced pure pyroxene, though the first trial was strongly dominated by an augite. The second attempt produced an augite and an olivine group mineral. The students believed that annealing helped increase the abundance of augite, though based on color alone, the annealing clearly oxidized the products.

Attempts were made to make the pyroxenes augite and enstatite using the Zybek reactor. Target compositions were selected based on the pyroxene phase diagrams that would only have pyroxene on the liquidus down to the solidus. It should be noted that phase relationships in the pyroxene family are complex and slight departures from ideal conditions can have significant effect. The first two attempts at augite developed obvious layering and three minerals were observed: augite, cristobalite, and quartz. The abundance of augite in the two attempts was 94.3% and 66.5%. Precisely why these results occurred has not been experimentally explored.

Enstatite (orthopyroxene, Mg₂SiO₆) synthesis was a bit more ambiguous; instead of getting the specific mineral enstatite, the x-ray diffraction pattern gives a best fit for a mixture of protoenstatite 70%, clinoenstatite 22% and minor amounts of quartz (6%), and magnetite (1%). Protoenstatite is a high-temperature polymorph of Mg₂SiO₆. Combined, the two forms of enstatite total 92%.

While the target pyroxene dominates the product in each case, the presence of undesired phases can present a significant problem to use of the material in a simulant. This is especially true if the undesired phase is crystalline SiO₂, which is the formula for both cristobalite and quartz. As noted earlier in the discussion of safety, in respirable form, these SiO₂ minerals constitute a significant health risk.

5.4.2 Anorthite

The mineral plagioclase is a solid solution of two end-member molecules, anorthite - CaAl₂Si₂O₈, and albite - NaAlSi₃O₈. The ratio of the two molecules is expressed as the percentage of the anorthite end-member. A crystal with An₉₀ is a crystal in which 90% of the molecules are the calcium end-member and 10% are the sodium end-member. The rocks of the lunar highlands typically contain plagioclase with >An₉₀. The authors have not been able to identify a practical terrestrial source of plagioclase with >An₉₀.

Wilson and Weinstein were successful in synthesizing two anorthites, An₁₀₀ and An₉₆. Both were made in very high purities; the only significant nonanorthite constituent being some local glass having the same composition as the mineral. Three runs made a total of approximately 20 kg of anorthite in three boules. One boule was slabbed and three locations (top, middle, interface between molten/powder) studied by SEM and x-ray diffraction. SEM analysis showed no inclusions of quartz, alumina, or CaO at levels above 1 part in 250,000.

6. PRODUCTION OF THE NU-LHT SERIES

6.1 Acquisition of Starting Material

Four trips were made to the property of the Stillwater Mining Company in Nye to collect bulk rock; the trips were November 16–17, 2006, October 17–19, 2007, September 21–24, 2008, and September 28–30, 2009. The primary reason for each trip was to collect bulk quantities of rock to be used for simulant manufacture. In addition to the collection of rock, each trip was also used to study the local geology, mine, and mill operations with the intent of optimizing the use of this critical resource. The trips were also used to educate nongeology personnel involved in simulant production or simulant use about relevant mining and milling technologies.

Materials were collected from the mine waste-rock pile, along road cuts above the primary mine portal, the Mouat/Mountain View Chromite properties, and from the mill sand tails stream. These materials formed the bulk of the material used in the NU-LHT series simulants.

A major objective in the collection process was to minimize the amount of secondary, hydrothermal alteration minerals introduced into the simulant manufacturing process. The alteration characteristics of the ore system at Stillwater are spatially variable, resulting in useful rock being mixed in with undesirable rock. It was found that nongeologists were generally unable to rapidly learn how to discriminate between desirable and nondesirable rocks. Therefore, each rock collected for simulant manufacture was hand selected personally by, or under the close supervision of, the geologists Doug Stoeser, Christian Schrader, or Doug Rickman. It should be noted that this hand selection process resulted in a significantly higher quality product, but also represents a very large percentage of the total cost per ton of the NU-LHT series simulants. It is avoidance of this cost element that, in part, motivates the need to develop mineral separation technologies for simulant production.

Material on the waste pile is heavily coated by a silty layer generated in the blasting process. Lithologies are also mixed in an approximately random manner. In the first collection effort on the waste pile, nothing was done to remove this dust. In a subsequent collection effort from the waste pile, the rocks were washed down with water from a tanker truck supplied by the Stillwater Mining Company (fig. 16). Removal of surface dust facilitated the proper identification of suitable material and allowed collectors to more readily and accurately separate the rock into several categories, i.e., clinopyroxene rich, orthopyroxene rich, and anorthosite dominant rocks.

Collections done from outcrop were essentially single lithologies at each collection location, which minimized the need for sorting by rock type. Material collected elsewhere did not need washing before collection.



(a)



(b)

Figure 16. Selecting and collecting rock: (a) From the waste pile of the mine. In the middle ground, behind the bin, one of the mine personnel is washing rock to make identification of acceptable material easier (note the use of personal safety equipment), and (b) from a road cut above the mine where norite is being collected. The small rocks visible in this photo are not optimally sized but are of good mineralogical quality.

Materials collected on the mountainside were transported by pickup truck to the mill site, where it was transferred for shipping. Shipment was done in either 55-gal steel drums or in plastic-lined aluminum cages ($\sim 1 \text{ m}^3$) made from salvaged liquid-chemical containers from the mill (figs. 16 and 17). These bins were capable of holding approximately three-fourths of a metric ton of coarse rock. Figure 18 shows the single lithology inside the bins.



(a)



(b)

Figure 17. Loaded bins: (a) Transferring to staging and (b) in the staging yard.

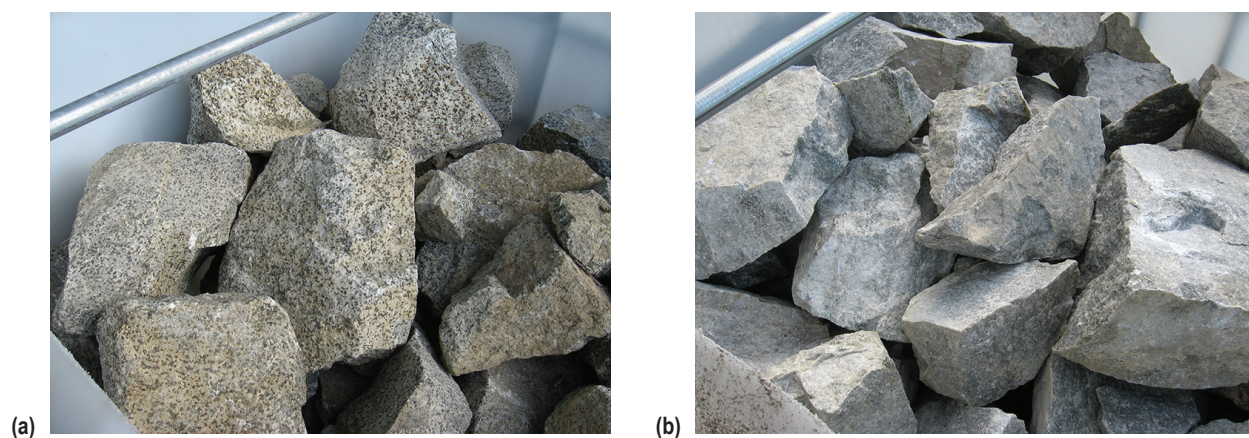


Figure 18. View inside loaded 1 m bins, each bin contains a single lithology: (a) Road norite and (b) anorthosite. Large rocks are preferred because it minimized the amount of hand labor in picking the sample up at the mine and minimizes the ratio of weathered to unweathered rock.

In addition to the rock, the company also provided 55-gal drums filled with mill sand. The mill sand is the fine-grained material left after the valuable minerals have been separated from the ore brought into the mill. It has several uses for lunar simulant production. It has already been crushed and ground to a consistent size distribution. It has a very uniform bulk composition due to the nature of the ore deposit and the mining method. It is also a reasonable mineralogy for many simulant design purposes. This material could be used as a simulant for some test purposes but a user would need to be aware that it does contain traces of the chemicals used in the mill's flotation processes. This material also has higher levels of hydrothermal alteration minerals than most of the rock in the Stillwater Complex. However, the mill sand was primarily used for a feedstock for the production of melt products, which eliminates these problems. This can strongly affect tests such as H_2O generation, melting points, and probably abrasiveness. The amount of material collected each trip varied from approximately 2 ton to more than 15 ton. Inventory collected on the November 2006 trip is listed in table 1.

Table 1. Inventory of material obtained from the Stillwater Mine in the November 16–17, 2006, trip.

Component	Amount (lb)
Anorthosite	800
Orthopyroxenite	450
Poikilitic harzburgite	450
Norite	2,300
Cpx-norite/ gabbro-norite	640
Chromite	450
Mill Sand	700
Total	5,790

The September 21–24, 2008, trip obtained 30,000 lb of rock and 3,000 lb of sand.

In this effort, the assistance of mine employees was absolutely essential. In addition to their personal labor, they also provided their personal vehicles to transport loaded bins of rock to the shipment staging point (fig. 17). In this effort special note should be made of the help of David Ryckman, Jeff Hughes, and Matt McManamen. Shipment from the mine property was by commercial contract carrier using flatbed trucks to the USGS facility in Denver, CO. Stillwater Mining provided the forklift and other logistical support to enable this.

Only material from Stillwater was used for the NU-LHT-1M prototype. Other materials were used in subsequent generations of the NU-LHT series. These materials were purchased commercially or were received as donations. A complete discussion is described in “Geologic Feedstock Development for Lunar Regolith Simulants” by Douglas B. Stoeser, Stephen A. Wilson, Douglas L. Rickman, and Michael A. Weinstein (in preparation). The following are excerpts from that report:

Olivine—There are only two companies in the United States that produce commercial olivine, Unimin Corporation that mines olivine in North Carolina and Olivine Corporation that mines olivine from the Twin Sisters Dunite in Washington State. Material from the Twin Sisters deposit was used in this work.

Ilmenite—For the LHT simulant series, beach sand ilmenite was obtained gratis from Iluka Resources. Subsequent work (in review) by Caroline-Emmanuelle Morisset (Canadian Space Agency), Marie-Claude Williamson (Geological Survey of Canada), and Victoria Hipkin (Canadian Space Agency) has shown that ilmenite from the Mirepoix, Sanford Lake, and Degrosbois Fe-Ti deposits in Quebec, Canada, would have been strongly preferable. These sources produce ilmenite free of hematite exsolution. Synthetic ilmenite can also be purchased.

Merrillite—Merrillite is very rare on Earth and is not available as a synthetic substance. The mineral whitlockite is highly similar to merrillite. A synthetic form of whitlockite is sold as beta-tricalcium phosphate (β -TCP). It is chiefly manufactured as a specialized dental material and is very expensive, being typically around \$1,500/kg. Alternatively, alpha-tricalcium phosphate, a polymorph of β -TCP, is also available commercially and probably feasible to make in the lab.

Apatite—Low grade, natural gem fluoroapatite was added as a minor component to the NU-LHT-2M simulant. Natural apatite, unlike synthetic, also has the advantage of having some amount of rare Earth elements.

Pyrite—Natural pyrite was added as a substitute for the troilite found in the lunar regolith.

6.2 Initial Processing in Denver

Upon arrival at the USGS, the material from each drum or bin was removed, washed if needed, and divided into two groups based on rock size (fig. 19). Samples $<10\text{ cm}^3$ were placed in

* Mining particles which can pass through a given sieve size are referred to as being ‘minus’ that sieve. Thus, minus 2 cm particles will pass a screen with openings 2 cm square.



Figure 19. Doug Stoeser with typical material received at the USGS facility in Denver from the Stillwater mine. Shipments used both 55-gal drums, as in this case, and 1 m³ containers made at the mine site from containers used to ship milling chemicals. These rocks are dominantly norite and anorthosite and have not yet been washed.

an ‘immediate processing’ pile. Those rocks $>10^3$ cm were reduced in size by hand using a sledge hammer. Each rock was also checked for suitability and inappropriate material rejected. Prior to grinding, all material was dried for 16 hr in a forced-air drying oven operating at 38° C (100° F). Also prior to grinding, lithologic samples were taken for petrographic characterization by thin section analysis.

After drying, material was mechanically crushed using a 6-in jaw crusher to produce minus 2 mm particles (fig. 20).^{*} Output from a jaw crusher will have a significant fraction that is larger than the output setting. Therefore output of the crusher was fed through a 2 mm vibratory screen and oversize was fed back through the crusher.

For analysis of major and minor elements, a split was taken from each rock type after crushing. The procedures necessary to assure a valid sampling for this purpose are beyond the scope of this TM. For guidance on this topic, the simulant producer should consult with someone familiar with geochemical sampling of whole rock or a relevant text.

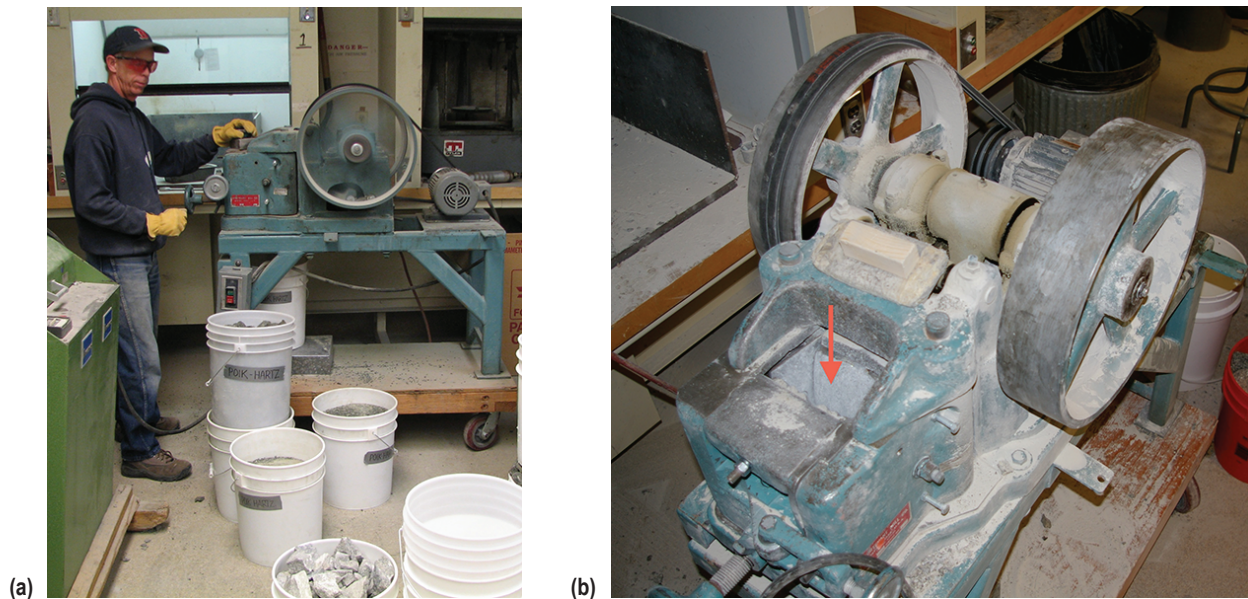


Figure 20. Jaw crusher: (a) Steve Wilson operating the 6-in jaw crusher. Material in the foreground bucket has yet to be run through the crusher. Material in the two buckets on the floor have been through the crusher, and (b) overhead oblique view of jaw crusher. The arrow marks the moving jaw, which is inside the in-feed opening of the machine.

The chemical information as normative mineralogy was used to specify the mixing ratios used to produce the simulants. Note that the use of normative mineralogy can only be used where the normative mineralogy of the source rock adequately matches the modal mineralogy. Petrologists have several ways of measuring the modal mineralogy of rocks; as there are numerous subtleties involved in such measurements, it is prudent to have a specialist superintend such work. For the Stillwater materials used in this work, the normative and modal mineralogy are reasonably matched.

Analysis of the starting rock is especially critical for a simulant made from more than one feedstock rock. A wide range of simulant compositions can be achieved if multiple, different feedstocks are available and mixed. This is a major advantage for both the producer and the user. Also, it may be possible to meet a single design and specification by more than one combination of the same end members. This happens because the feedstocks are themselves complex systems of similar minerals, differing in part in the ratios of the minerals. Also, the producer must be aware that the different feedstocks contain different amounts of nonlunar phases and there are detailed differences in the exact mineralogy of each feedstock. For example, if one starts with the Stillwater norite and wishes to increase the total plagioclase, it is possible to add Stillwater anorthosite. But compared to the norite, the anorthosite from Stillwater has lower values of calcium in the plagioclase and has different relative abundances of clinopyroxene and orthopyroxene. Thus, changing one attribute of the simulant, the amount of plagioclase, also changes the calcium abundance and the pyroxene ratios.

6.3 Grinding Experiments

Because a specific particle size range was desired, grinding time experiments were performed to determine the effect of grinding time on particle size on each rock type collected. In one experiment, 50 kg aliquots of each rock type were placed in a ceramic-lined, 30-gal (0.11 m³) ball mill equipped with 70 kg of 1 inch diameter aluminum oxide ceramic grinding balls (fig. 21). Over the period of 12 hr, a series of 10 samples were collected from the ball mill and sieved using stainless steel sieves in an automated sieving device (Rotap). The contents of each sieve size were weighed and the mass of material collected on each sieve was recorded (fig. 22).



Figure 21. Ball mill with two 5-gal buckets of aluminum oxide ceramic grinding media in the foreground. Rock to be ground and the grinding media are poured into the port visible on the top quadrant of the rotating drum.

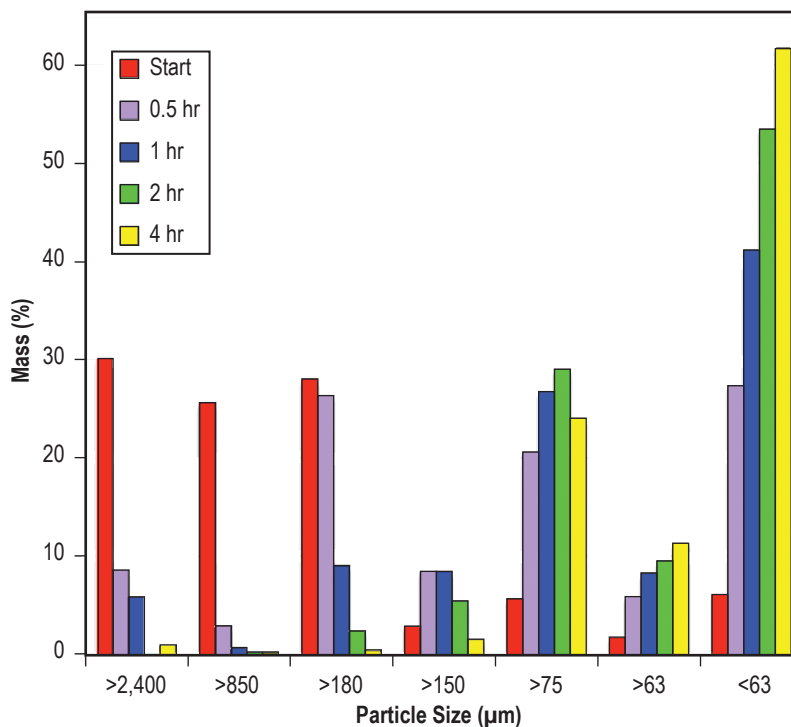


Figure 22. Particle size as a result of different grinding times, minutes, in the 30-gal ball mill with aluminum oxide ceramic media for Stillwater norite.

In another experiment the particle size versus time versus starting mass of rock loaded was evaluated. Another 24-hr grinding experiment showed that particle size versus time is dependent on the ratio of grinding medium (ceramic balls) to crushed rock material and the particle size of the starting material. One experiment also evaluated the reproducibility of the ball mill processing (fig. 23). The data show that easily measured variation in particle size distributions exists between batches. Whether or not this is significant depends on the application of the simulant. The authors have insufficient data to indicate whether the measured variation is typical in such a situation. It was felt that for the intended purpose of the simulant, the amount of observed variation between rock types was small enough that it could be ignored.

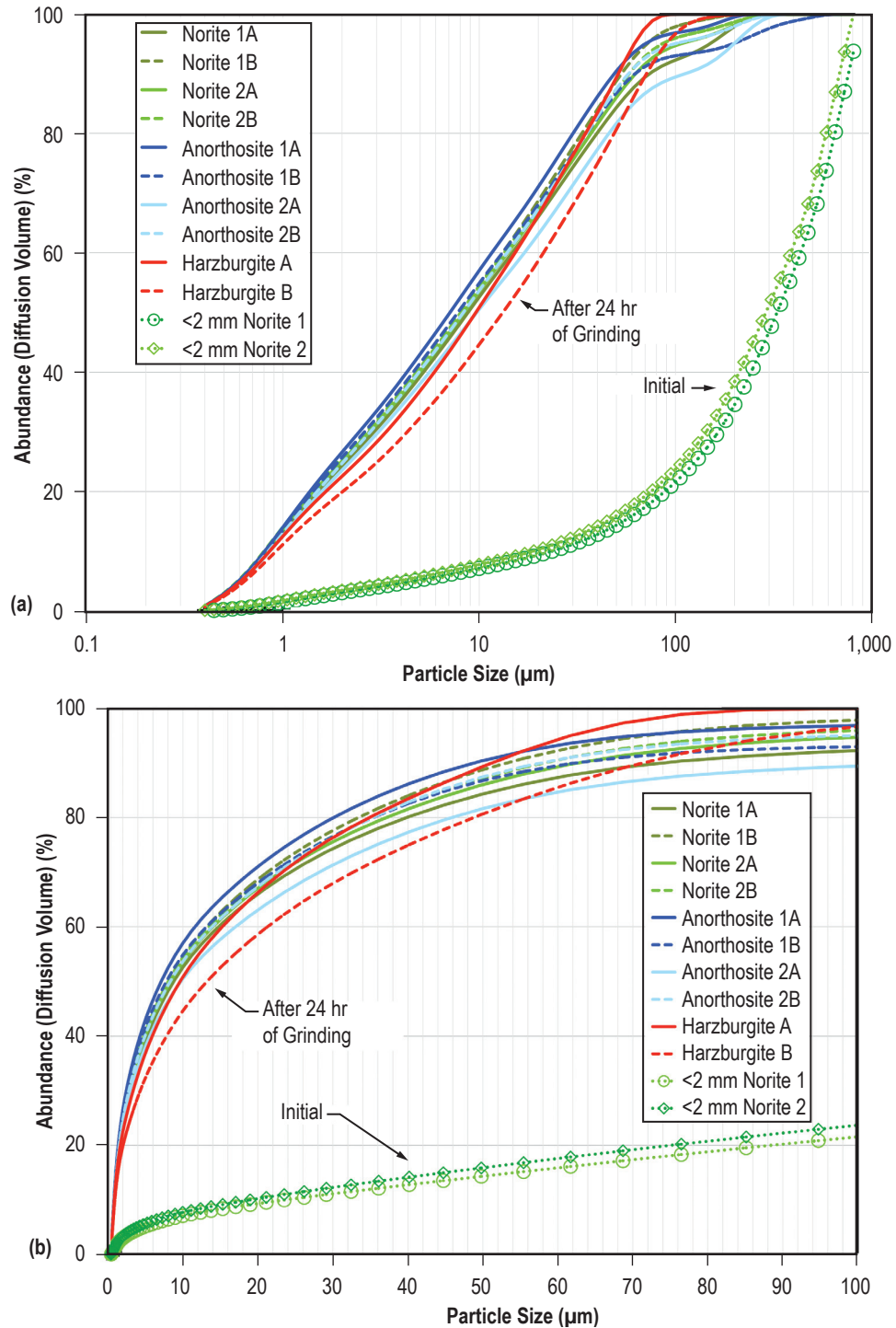


Figure 23. Reproducibility of particle size distributions as a function of rock type after 24 hr of grinding. Two samples of norite and two samples of anorthosite were each ground twice. A single sample of harzburgite was ground twice. For comparison, the distributions of the starting norite samples are also shown. Data were measured by a Coulter LS laser diffractometer: (a) Logarithmic plot of data to 1,000 μm and (b) linear plot of data from zero to 100 μm . The vertical axis of both graphs is the cumulative volume percent.

Examination of figure 23 shows that all three rock types generate a similar particle size distribution pattern. The relative amount of fine-grained material is fairly consistent for all three rock types once the particle size is below 50 μm . Calculation of the ‘dust component’ of these materials using a 10 μm upper limit reveals that $\approx 50\%$ of the ground material could be considered a dust. This suggests that with the tested grinding equipment, the creation of a dust simulant material with the correct proportional distribution of rock types is possible by removing the $>10 \mu\text{m}$ fraction or extending the grinding period from 24 to 36 hr.

Grinding tests were also done for two synthetic glassy components—pseudo-agglutinates and high-quality glass. The test objective was to evaluate how the glasses would survive the grinding process. The mixing composition material from the Stillwater mine listed in table 2 was combined in the ball mill as two 22-kg (50-lb) batches. The glass material added to each grinding run was 9-kg (20-lb) pseudo-agglutinate and 2.2-kg (5-lb) high-quality glass. The first batch was ground for 60 min and the second batch for 15 min. The two batches are referred to below as the 60 batch and 15 batch.

Table 2. Crystalline rock mixture used in glass grinding test.

Component	%
Norite	37.5
Anorthosite	54
Harzburgite	7.5
Ilmenite	1

SEM analysis of the 15- and 60-min samples reveals that the pseudo-agglutinates were ground so fine they were not observed in the single sample taken from each batch. The high-quality glass was observed in both samples with a higher percentage of larger fragments observed in the material ground for just 15 min.

As a result of the grinding experiments, the USGS designed a scoring protocol to estimate the grinding time necessary to achieve a specific particle size distribution. Examination of particle size distribution profiles for Apollo 16 samples indicated that a grinding time of 2–3 hr would be sufficient to produce a simulant material with a top end particle size of approximately 1 mm.

The design document for the simulant being produced specifies the ratio of feedstock rock types used, which for NU-LHT-1M is given in table 2.⁶ Once the grinding time experiments were completed, aliquots of each rock type were combined as per the design in batches of 50 kg and ground for 16 hr. This time provided a reasonable abundance of minus 100 μm particles without destroying all of the coarser particles.

6.4 Mixing and Splitting

After grinding, material was removed from the ball mill and transferred to a 10-ft³ (0.28-m³) cross-flow V-blender (fig. 24). A total of 300 kg of material could be blended at one time.



Figure 24. Steve Wilson standing beside the cross-flow V-blender.

Following a 6-hr blending interval, the blender was emptied into a series of 5-gal plastic buckets. Subdividing the blended material into representative aliquots was performed using a USGS-designed spinning riffler (fig. 25), in a two-step process. In the first step (coarse splitting) material from the V-blender was transferred to a 2-ft³ (0.056-m³) hopper that delivered powdered material onto a rotating circular platform operating at 10 rpm. (When trying to design or scale a splitting process, the relationship between volume and mass for the simulant must be known. The values determined by Zeng et al.⁴⁵ for NU-LHT-2M are between 2.05 and 1.36 kg/m³. Other simulants are comparable.) Positioned at the outer edge of the platform are 12 tapered V-splitters having a downward angle of approximately 60° (fig. 26). The V-splitters are held at a height of 45 cm

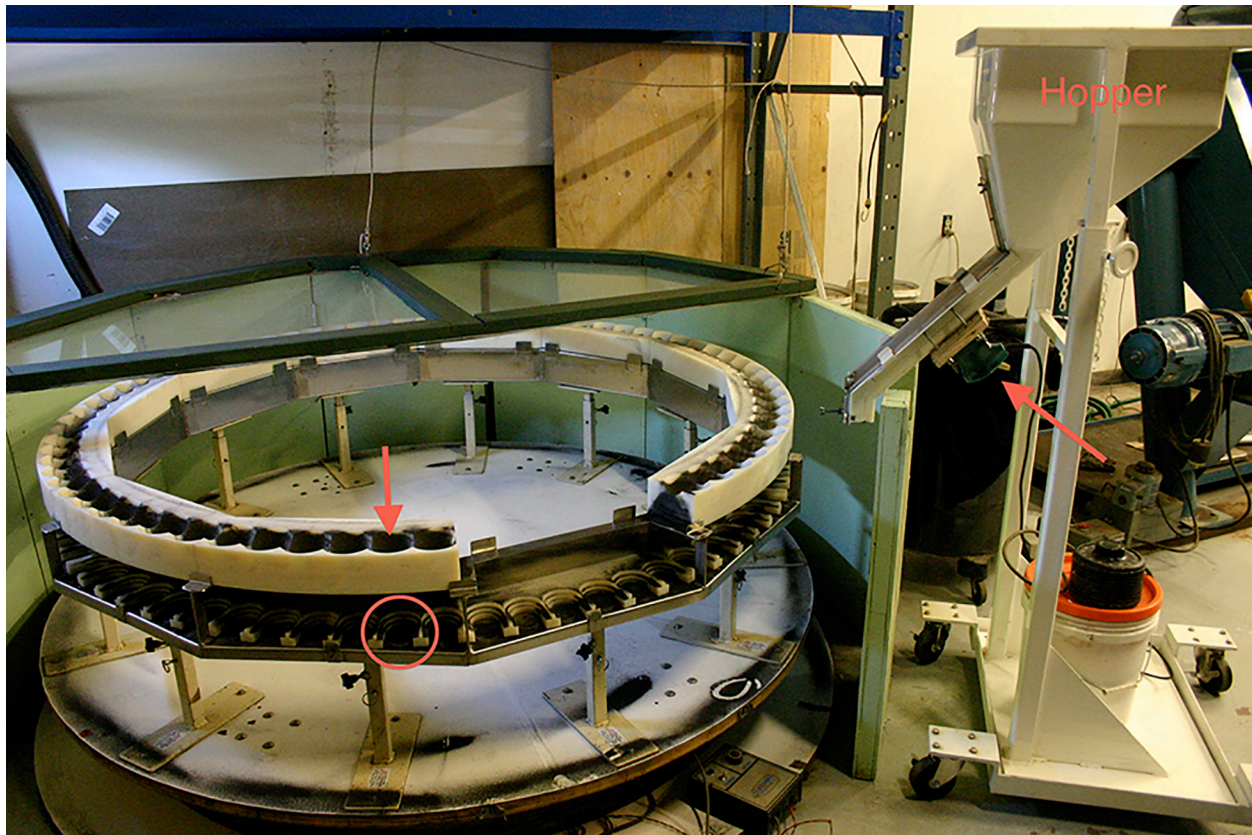


Figure 25. Rotating riffle splitter devised by Steve Wilson of the USGS. Note the palm sander attached to the hopper exit chute to provide vibration. In this configuration the machine will fill bottles. The left arrow points to mouth of a Delrin funnel. The circle below the arrow encloses a registration or holder for the bottle corresponding to the funnel. The drive mechanism for rotation is below the mounting table holding the support platforms. The hopper is pulled back from the splitter, one of the Delrin funnel blocks has been removed, and the dust containment wall and covering glass shield have been partially removed.

above the platform surface using stations equipped with a pin and screw-down clamp system. Prior to the start of the splitting step a set of two 5-gal buckets are positioned beneath each V-splitter, for a total of 12 buckets. Five-gallon buckets are readily available, inexpensive, and can hold between 23–30 kg of ground rock, which is as heavy as the operator of the splitter can safely move about. As each V-splitter unit passes beneath the exit chute of the hopper, ground material is distributed equally into the right and left 5-gal buckets. The rotation rate of the platform and the out-feed rate from the hopper are such that many rotations of the platform occur for each hopper load. A maximum of 270–360 kg of material may be subdivided in this manner, which is the output of the cross-flow V-blender.

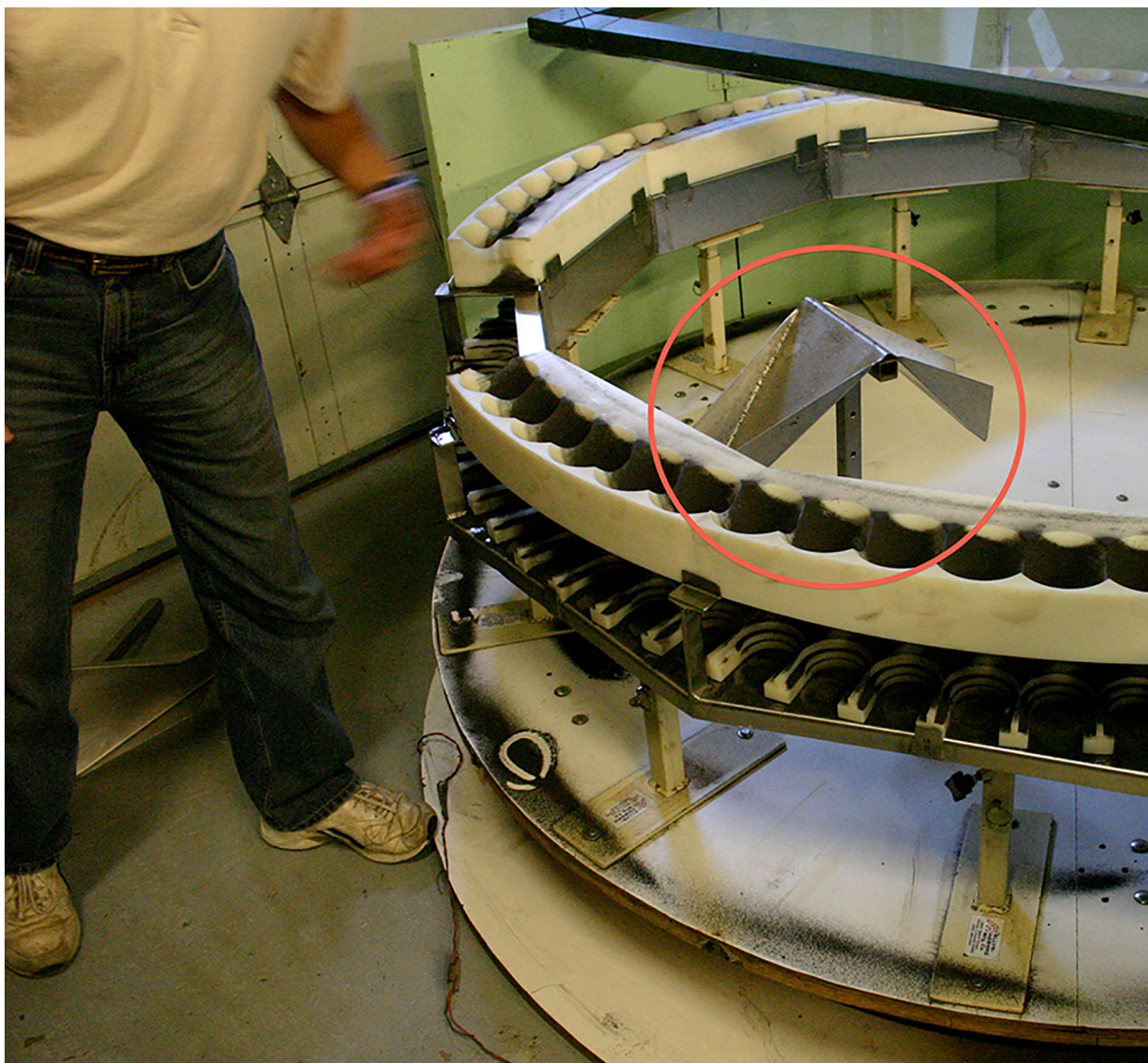


Figure 26. Detail of rotating riffle splitter showing a single V-splitter. The V-splitter is not installed in its operational position.

If additional aliquots of the same material are to be split, a fresh set of 5-gal buckets are placed at each splitting position and the process is repeated. When multiple splitting runs are required, individual splits from the same splitter position are labeled with the specific position and the run number (position, run) for example 1-1, 1-2 ...1-n ; 12-1, 12-2, ...12-n. This numbering system is critical for the proper splitting of material in subsequent runs. The material within a run has been blended and then split 12 ways, and to a high level of precision, the material in each of the 12 buckets from a splitting run is uniform. The differences between runs will exceed the differences within a run. The mass of each container is determined and then the containers are ordered in terms of decreasing mass.

The bottling step occurs after coarse splitting. The desired per bottle mass is multiplied by 72, the number of bottles in the final bottling run, to determine the required minimal per run bottling mass. There will be a range in sample mass per container, which will theoretically follow a normal distribution. To minimize the chance that the mass per container will fall below a target amount, the minimal bottling mass may be increased by 10%–20% at the operator's discretion. The minimal bottling mass corresponds to a number of 5-gal buckets. This number of buckets must be available to complete the final bottling run. This may require the combination of multiple sample buckets from multiple runs to obtain the required minimal per run bottling mass.

In the bottling stage, a series of 12 support platforms are installed which support a 12-section stainless steel splitting wheel. A six-position funnel block made from DuPont Delrin (polyoxymethylene) is added to each section of the wheel along with a six-position bottle holder block (figs. 24 and 25). When properly fitted, the splitting wheel with funnel blocks stands approximately 45 cm above the surface of the platform. The spinning wheel is then loaded with 72 bottles (caps removed). The hopper is filled with a specific bottle mass aliquot, the platform rotation initiated, the hopper valve adjusted to the proper height, and when necessary, the vibratory feeder initiated. Powder is dispensed from the hopper through the funnel block and into the respective bottles. The filling process is continued until the entire bottling mass aliquot is distributed. At the end of the bottling cycle, the bottles are removed from the wheel, the per bottle sample mass determined, and the lids attached.

During the bottling cycle, samples for analysis are removed from the final set of bottles at discrete intervals. These stratified-random samples are later subjected to chemical and physical characterization. This selection process provides a between-bottle homogeneity assessment which, when combined with within-bottle replicates, determines the sampling error in the final statistical analysis. For the NU-LHT series, simulants' chemical analysis of the selected samples was performed at the USGS and its contract laboratory. Total element concentrations were determined following a multiacid or acid-fusion sample decomposition (table 3).

Table 3. Major and trace element analysis of NU-LHT-1M. Data from the Preliminary Certificate of Information of NU-LHT-1M.

Element	Mean (wt. %)	Standard Deviation	Oxide	Mean (wt. %)	Standard Deviation
Al	12.9	1	Al ₂ O ₃	24.4	3.5
Ca	9.38	0.4	CaO	13.1	0.56
Fe (II)	2.55	0.01	FeO	3.3	0.012
Fe _{TOT}	3.35	0.17	Fe ₂ O ₃ T	4.79	0.24
K	0.07	0.01	K ₂ O	0.08	0.01
Mg	5.12	0.29	MgO	8.5	0.48
Na	1.06	0.02	Na ₂ O	1.43	0.03
P	<0.01	–	P ₂ O ₅	<0.02	–
S _{TOT}	<0.05	–	–	–	–
Si	22.26	0.1	SiO ₂	47.62	0.21
Ti	0.2	0.01	TiO ₂	0.33	0.02

Table 3. Major and trace element analysis of NU-LHT-1M. Data from the Preliminary Certificate of Information of NU-LHT-1M (Continued).

Element	µg/g	Standard Deviation	Element	µg/g	Standard Deviation	Element	µg/g	Standard Deviation
Ag	<2	–	Hf	2	<0.05	Sm	0.25	0.05
As	<30	–	Ho	0.08	0.01	Sn	<1	–
Ba	23	1.3	In	<0.2	–	Sr	106	5
Be	<1	–	La	2.95	0.3	Ta	<0.5	–
Bi	69	9	Li	2.76	0.21	Tb	0.06	0.01
Cd	<0.05	–	Lu	0.05	0.005	Th	0.31	0.03
Ce	17.1	2.3	Mn	600	23	Tl	<0.5	–
Co	29.9	4.1	Mo	3.6	2.5	Tm	<0.05	–
Cr	662	62	Nb	4.93	0.98	U	0.12	0.03
Cs	0.1	0.01	Nd	1.42	0.08	V	51.7	5
Cu	38.3	4.1	Ni	323	14	W	<1	–
Dy	0.48	0.03	Pb	1.54	0.23	Y	2.65	0.42
Er	0.25	0.02	Pr	0.38	0.01	Yb	0.3	0.005
Eu	0.19	0.02	Rb	1.69	0.12	Zn	26.8	3.4
Ga	12	0.67	Sb	0.05	0.01	Zr	74.6	4.1
Gd	0.3	0.3	Sc	10.5	1.4			
Ge	<1	–	Se	<1	–			

Material prepared in the manner described above was later designated as NU-LHT-1M.

While chemical analyses are necessary, the mineralogy is actually what controls the physical properties of the simulant. To illustrate, carbon can be both graphite (one of the softest minerals known) and diamond (one of the hardest minerals known). A modal analysis of the NU-LHT-1M material by SEM provided the information in table 4. One observation made in the process of analysis was that the high-quality glass could not be readily discriminated from the pseudo-agglutinate glass. Also important for the user is to know the abundances of nonlunar phases. As the nonlunar phases are not abundant, presumably, some special effort may be needed to quantify their abundances.

Table 4. Modal analysis of NU-LHT-1M by particle size fraction. Values are estimated to be within 15% of the stated value.

	Total NU-LHT-1M	<75 μm	75–150 μm	150–400 μm	>400 μm
Wt. % fraction	–	30.7	28.7	34.8	5.8
Glass	39.88	32.48	28.86	55.32	40.97
Plagioclase	40.74	46.85	47.16	29.66	43.13
Olivine	2.16	2.28	3.35	1.23	1.29
Orthopyroxene	4.39	4.65	4.71	3.58	6.21
Clinopyroxene	7.71	6.7	8.27	8.09	7.92
Ilmenite	0.93	0.96	2.2	–	–
Chromite	0.04	0.05	0.01	0.06	0.09
Sulfide	0.03	0.11	–	–	–
Phosphate	–	–	–	–	0.01
Subtotal	95.88	94.08	94.56	97.94	99.62
Nonlunar Other Minerals					
Albite	2.97	5.18	2.86	1.58	0.09
Quartz	0.18	0.1	0.21	0.21	0.22
FeOx	0.14	0.07	0.07	0.27	0.06
Amphibole	0.2	0.57	0.09	–	–
Talc	0.34	–	1.2	–	–
Carbonate	0.22	–	0.77	–	–
Subtotal	4.05	5.92	5.2	2.06	0.37
Total*	99.93	100	99.76	100	99.99

*Totals rounded up to 100.

6.5 Preparation of NU-LHT-2M

Based on what was learned in the creation of NU-LHT-1M, a second version was made, NU-LHT-2M. Source materials were collected for this in much the same manner as already described above. In general, rocks with a mass greater than approximately 20 kg were reduced at the collection site using sledgehammers. Rocks ranging in mass from 2 to 20 kg were transferred to plastic-lined aluminum cages. Upon arrival at the USGS, rock samples greater than 5 kg (30 cm²) were reduced in size to 1 kg using sledgehammers. The rock material was then crushed to less than 2 mm using a 6-in mechanical jaw crusher. Crushed material was sieved through a 2-mm vibrating sieve and material >2 mm was reprocessed through the jaw crusher. At the end of the crushing cycle material, <2 mm was then transferred to ceramic-lined ball mill for final size reduction.

In order to produce homogeneous simulant material with the desired particle size distribution containing agglutinate, a separate set of grinding experiments was performed using the <2 mm starting material. An optimal final particle size distribution was obtained when products from a 20- and 45-min grinding period were combined. Using these relatively short grinding intervals had several advantages. The first was that agglutinate material added at the start of the grinding

period (20 min) maintained a significant proportion of its structural integrity during the grinding process. Material from the 45-min grinding period contained less of the original agglutinate material but smaller agglutinate fragments still displayed a mixture of melted and crystalline material. The combination of material from the two grinding periods produced agglutinate material covering the entire particle size distribution range. A second advantage of adding agglutinate at the beginning of the grinding period is the improvement in material homogeneity relative to combining components during the blending stage.

Blending was performed at the USGS using a 10-ft³ (0.28-m³) cross-flow V-blender. Optimal blending conditions utilized a single batch approach where the entire sample amount was blended in a single run. When the volume of material exceeded the capacity of the blender, a modified blending process called a ‘continuous batch’ approach was used. In this blending process, the blender is initially filled with material sufficient to fill the blend within 75% of its blending capacity. After the 6-hr blending period, the blender is stopped and half the contents of the blender are emptied into a series of containers. The blender is then refilled with a second aliquot of the same material and the blending cycle repeated. This process of removing and refilling the blender is repeated until all the material is blended.

For process control, an important concern when targeting a specific particle size profile is how to evaluate a size distribution relative to a target value. For production of NU-LHT-2M, the following methodology was used:

- A size distribution was obtained from a Coulter LS laser diffractometer owned and operated by the BoR in the Denver Federal Center.
- Integration of the area beneath the particle size distribution profile was deemed the most direct and accurate measure. This measurement process may be subject to incorrect results when a normal size distribution is compared to a multimodal condition, but for most cases, this bias is not observed.
- In the preparation of the materials, an automated area calculation option was not available so an alternative approach was used. In this approach, the number of particles covering a specific size range was assigned a value (%Rel) corresponding to the fraction of particles in that size range relative to the total number of particles measured. This relative percent amount was then multiplied by the particle size (in microns) associated with that sized range. This process was performed for all 75 size ranges measured and the summation of those values is considered representative of the particle size distribution. This value was identified as the particle size score (PS score) for that analysis.

The following summarizes the major processing differences between NU-LHT-1M and NU-LHT-2M:

- Particle size—NU-LHT-2M had significant dust component (8%–10%) added to the starting mass. The dust size glass contained 50% glass. Size analysis of NU-LHT-1M indicates that 13% of total volume is <20 µm. Size analysis of NU-LHT-2M indicates 25% of the total volume is <20 µm.

- **Composition**—Mineralogical composition of NU-LHT-2M is a better match to the targeted lunar regolith.⁶ This was in part achieved by using commercial olivine from the Twin Sisters, Washington, deposit instead of Stillwater harzburgite. The commercial olivine did not add the additional orthopyroxene, chromite, and alteration minerals inherent in the available Stillwater harzburgite. In addition, trace minerals (pyrite, whitlockite, and fluoro-apatite) were added to match those typical of the highlands regolith. Table 5 gives the relative amounts of the feed-stocks used in NU-LHT-1M and NU-LHT-2M.

Table 5. Relative abundance of inputs for NU-LHT-1M and NU-LHT-2M.

Component	Relative Abundance (%)	
	NU-LHT-1M	NU-LHT-2M
Norite	30	19.8
Anorthosite	43	42.4
Harzburgite	6.1	–
Olivine	–	5.32
Ilmenite	0.72	0.6
Pyrite	–	0.05
Whitlockite	–	0.1
Fluoro-apatite	–	0.05
HQ glass	3.96	4.5
Pseudo-agglutinate	15.9	27.1

- **Particle texture**—In contrast to NU-LHT-1M, for NU-LHT-2M, a majority of agglutinate component was added during the blending stage (post grinding) to improve likelihood of agglutinate material maintaining original physical characteristics. Also, a limited quantity of glass spheres (<2 mm) were added to NU-LHT-2M during the blending stage.

APPENDIX A—RECOMMENDED SAMPLE PREPARATION PROCEDURES

It is recommended that the following procedures be used when obtaining sample aliquots from the original container of NU-LHT-2M. These suggestions are designed to mitigate any partitioning in the sample that may have occurred during sample transport or after the sample has remained on the shelf for a prolonged period of time.

- Prior to removing samples from the original container, it is recommended that the sample be mixed for a period of 15–20 min. Mixing can be achieved by gently rolling the container on its side using a standard roller mill or using a rotary agitation device (end-over-end mixing) similar to that recommended in Environmental Protection Agency method 1311, Toxicity Characteristic Leaching Procedure. Vigorous agitation should be minimized so that agglutinate material present in the sample is not reduced in size.
- When multiple sample aliquots are desired, it is recommended that the sample be split using a standard Jones type splitter. A Jones splitter is a device used to reduce the volume of a sample. It consists of a belled, rectangular container, the bottom of which is fitted with a series of narrow slots or alternating chutes designed to cast material in equal quantities to opposite sides of the device. The sample is split in a series of steps until the sample of optimal mass or volume is obtained. This type of device is available through catalogue companies (e.g., <www.humboldt-mfg.com> at a modest price (\$300–\$400).
- If a splitter device is not available, another sampling option is to use the cone and quartering technique. In this method, the contents of the container are emptied on to a flat surface (sheet of paper) in a cone configuration. The pile is initially split in half using a large spatula and that pile is again split in half using the spatula. This should leave a sample approximately one quarter of the original material. The sample remaining on the paper is mixed by tilting the edges of the paper and then the pile shaped once again into a cone. Subdividing/mixing the pile is repeated until the desired sample size is obtained.
- A sample thief may also be employed when a single sample is required. A sample thief is a tapered tubular device typically made of plastic, glass, or metal. The device is pushed into the solid sample using a rotating/twisting motion. The direction of the coring action should be along the longest possible axis of the container. Once the tube passes completely through the solid sample, it is gently removed using a constant twisting motion. Commercially available sample thieves may have a tube within a tube that allows the collected sample to be trapped within the thief, preventing any loss of sample during removal. The diameter of the tube and the cross-section depth will dictate the mass of sample obtained. It is imperative that samples collected from the entire cross section be obtained.

- One additional option for subdividing bulk powders is a sieving or microrotary riffler. This equipment takes powder and divides it between eight containers by rotating the containers beneath the exit chute of the powder reservoir. This spinning riffler approach is normally considered the optimal way to subdivide bulk powders. Commercial units (Quantachrome) are available for between \$4,500 and \$6,500.

A discussion of sampling techniques may be found in Jillavenkatesa et al.¹⁶

APPENDIX B—INDUSTRIAL HYGIENE REPORT ON BP-1

Appendix B contains pertinent pages from the Industrial Hygiene Report on BP-1.



May 12, 2010

T201003-1540

Greg Galloway, NE-S
NASA
Kennedy Space Center, FL

SILICA CONTENT, CRISTOBALITE, QUARTZ, AND TRIDYMITE AGGREGATE PRODUCTS (LIMESTONE/DOLOMITE, GRANITE/BASALT, SAND OR GRAVEL) BLACK POINT LAVA (BP-1)

REPORT SUMMARY:

Analytical results obtained from the laboratory indicate the presence of silica (cristobalite and quartz) in the material sampled. The handling, disturbance, demolition, and disposal of this material is subject to federal, state and local requirements as indicated below.

The IHA Industrial Hygiene Office performed bulk sampling of a simulated lunar soil Black Point Lava (BP-1) per your request. The purpose of the sampling was to characterize the silica content (cristobalite, quartz, and tridymite) in the simulated lunar soil in sixty containers located in the high bay of M7-1104/Multi-Payload Processing Facility. Ten random samples were obtained and submitted to a certified laboratory for analysis. Laboratory analysis results were received by this office on April 25, 2010 and are noted in Table 1 below.

The bulk samples were collected and submitted for analysis for the 3 polymorphs of crystalline silica-quartz, cristobalite, and tridymite using the NIOSH 7500 method with the analysis to be performed by XRD and reported as %-wt.



Figure 1 Simulated lunar soil (Black Point (BP-1) stored in M7-1104



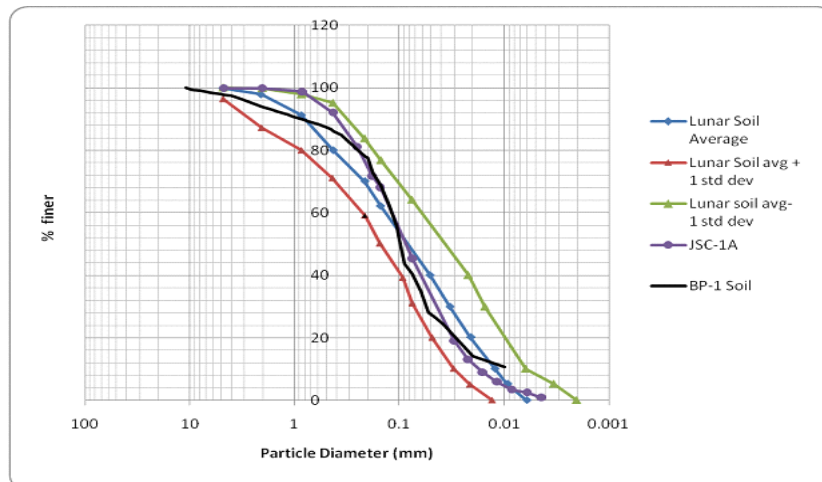
Figure 2 The simulated soil ranges from fist size rocks to fine powder.

TABLE 1

Silica/Cristobalite, Quartz, and Tridymite Content Simulated Lunar Soil Black Point (BP-1) M7-1104/Highbay			
SAMPLE NUMBER CONTAINER NUMBER	Cristobalite %-Weight	Quartz %-Weight	Tridymite %-Weight
01004017 BAG # 5	0.97	<0.5	<0.5
01004018 Bag # 7	1.2	0.52	<0.5
01004019 Bag # 12	1.0	<0.5	<0.5
01004020 Bag # 17	1.1	1.4	<0.5
01004021 Bag # 19	0.96	1.2	<0.5
01004022 Bag # 33	0.99	1.0	<0.5
01004023 Bag # 41	0.57	2.3	<0.5
01004024 Bag # 42	0.70	<0.5	<0.5
01004025 Bag # 48	2.9	<0.5	<0.5
01004026 Bag # 52	1.6	0.51	<0.5

<=Indicates below the limit of detection for the analytical method used

Particle Sizing. To help understand why the Black Point (BP-1) soil behaves so much like lunar soil, its particle size distribution was measured by the Granular Physics Lab by dry and wet sieving using a Retsch sieve shaker using pan sizes 9 mm to 10 μ m. Once wet sieving was finished, the samples (including the catch bucket of water) were dried in a convection oven at low heat and the particulate mass of each was measured. The soil was also measured on a Fine Particle Analyzer (FPA), which uses a gas dispersion technique with a telecentric microscope and strobed backlighting to rapidly image millions of individual particles down to just a few microns in size.



CONCLUSIONS

Analytical results indicate that silica content (cristobalite, quartz, and tridymite) in the simulated lunar soil is present as shown in Table 1. The potential for personnel exposure to the crystalline silica found in the simulated lunar soil depends on the amount of disturbance the soil is subjected to by personnel and mechanical equipment including the robots during the mining competition, weather conditions, and enclosure design.

Based on the physical properties of the black point lava and the particle size distribution, the material has a high potential to become airborne. Based on the properties of the BP-1 and activities to be performed during the event, a reasonable potential exists for participants to be exposed to silica levels in excess the occupational exposure limits. As the judges will be expected to have more frequent and, potentially, longer duration entries into the sand box, their silica exposures are expected to be greater.

The PPE recommendations for contestants (college students) are based on control of short term acute exposure to the black point lava material. Short term, acute exposure to silica would not be expected to have long term health effects, but may irritate the nose, throat, and respiratory tract by mechanical abrasion. The potential exist for the judges to be exposed to the BP-1 material on a recurring basis. Chronic exposure to respirable dust containing crystalline silica in excess of the OSHA PEL is related to an increased risk of developing silicosis. Since there is potential for recurring contact with the BP-1 material, and they are expected to have the highest exposures of the participants, a higher level of respiratory protection is recommended for use by the judges. Personnel protective equipment recommendations are provided in Table 2.

The MSDS indicates that inhaling respirable dust and/or crystalline silica may aggravate existing respiratory disease(s) or dysfunctions. In addition, exposure to dust may aggravate existing skin and eye conditions. The hazards associated with the BP-1 material must be communicated to those performing work with the material.

RECOMMENDATIONS

The IHA Industrial Hygiene Office recommends that personal protective equipment and controls be used to ensure adequate protection of the workers and adjacent, unprotected personnel until measurements are obtained to characterize the potential for exposure. Recommended controls are to exclude personnel from the area during the mining competition, a decontamination area with HEPA vacuum for robots, and a handwash area that can be utilized when exiting the box. A portable eye wash should be available at the site. Depending on the wind conditions, the sides of the tent can be used to control the amount of dust which becomes airborne. Personnel protective equipment recommendations are provided in Table 2 to reduce exposure for those individuals who are entering the sandbox.

TABLE 2 PPE Recommendations for Lunabotics Preparation and Competition		
<u>Exposure Group</u>	<u>Exposure Frequency/Duration</u>	<u>Recommended Personal Protective Equipment</u>
Contestants (College Students)	Short Term, As required estimated to be <45 minutes per day	Filtering Facepiece with N95, Tyvek suits and booties (or equivalent), dust goggles
Judges (NASA)	Several times per day, Additional exposure possible through continued work with sandbox	Half Face Respirator with P100 cartridge, Tyvek suits and booties (or equivalent), dust goggles

The use of respiratory protection for judges must comply with OSHA respiratory standard 1910.134 to include medical certification, fit testing and training. There is a duty under the OSHA Hazard Communication standard to communicate the potential hazards to all individuals that will be entering the lunar sand box. Prior to entrance into sandbox each individual should receive a pre-task briefing and a MSDS should be provided for review.

To characterize airborne exposure levels during the filling of the simulated lunar sand box, competition events, and tear down of the arena personnel air sampling will be performed to determine the potential exposure to respirable dust to include crystalline silica.

If you need additional information or have any questions concerning this report please contact this office at 867-2400.

A handwritten signature in cursive script that reads "Lisa Brown".

for
John Sherwood
Industrial Hygiene, IHA-022

RAP:JWS:dlg

Attachment:

1. HEALTH HAZARDS FOR CEMEX AGGREGATE PRODUCTS

cc: Michael Cardinale, TA-B1A

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14. ABSTRACT The manufacture of lunar regolith simulants can use many technologies unfamiliar to the aerospace industry. Many of these technologies are extensively used in the mining industry. Rock crushing, grinding, process control as a function of particle size, as well as other essential concepts are explained here. Notes are provided on special considerations necessary, given the unusual nature of the desired final product. For example, wet grinding, which is an industry norm, can alter the behavior of simulant materials. As the geologic materials used for simulants can contain minerals such as quartz and pyrite, guidance is provided regarding concepts, risks, measurement, and handling. Extractive metallurgy can be used to produce high-grade components for subsequent manufacture, reducing the compromises inherent in using just rock. Several of the components needed in simulants such as glasses, agglutinates, and breccias are simply not available or not reasonably matched by existing terrestrial resources. Therefore, techniques to produce these in useful quantities were developed and used. Included in this list is the synthesis of specific minerals. The manufacture of two simulants, NU-LHT-1M and NU-LHT-2M, is covered in detail.					
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